LABORATORY EXERCISES

TO ACCOMPANY

FIRST PRINCIPLES OF CHEMISTRY

BROWNLEE · FULLER · HANCOCK · SOHON · WHITSIT

REVISED EDITION

Chemistry
Room 139
Mr Bailey

No 24

Ruth Lowell
Room 300
L. H. S.

Digitized by the Internet Archive
in 2025

https://archive.org/details/laboratoryexerci0000brow

LABORATORY EXERCISES

TO ACCOMPANY

FIRST PRINCIPLES OF CHEMISTRY

BY

RAYMOND B. BROWNLEE
STUYVESANT HIGH SCHOOL

ROBERT W. FULLER
STUYVESANT HIGH SCHOOL

WILLIAM J. HANCOCK
ERASMUS HALL HIGH SCHOOL

MICHAEL D. SOHON
MORRIS HIGH SCHOOL

JESSE E. WHITSIT
DE WITT CLINTON HIGH SCHOOL

ALL OF NEW YORK CITY

REVISED EDITION

ALLYN AND BACON
Boston New York Chicago

COPYRIGHT, 1908 AND 1917, BY RAYMOND B. BROWNLEE,
ROBERT W. FULLER, WILLIAM J. HANCOCK,
MICHAEL D. SOHON, AND JESSE E. WHITSIT.

EDR

PREFACE

This Laboratory Manual is designed to accompany the authors' "First Principles of Chemistry." It is, in some measure, founded on Handbook 21 of the State Department of Education of New York, which was prepared by the authors in the spring of 1905, and which met with such success as to lead to the writing of the "First Principles of Chemistry."

Such of the experiments from the Handbook as appear in the present Manual have been carefully revised and improved where experience has shown this to be desirable. A number of other experiments have been added in order to give greater freedom of selection, and to provide fully for such schools as are favored with ample time for laboratory work. The authors believe that these exercises will be found to furnish a typical range of experiments suitable for an elementary course. Though practical and industrial applications receive considerable attention, yet a sound knowledge of the fundamental facts and principles of the science is considered of most importance to the beginner, since it is only through painstaking labor along theoretical lines that the achievements of industrial chemistry have been obtained.

It is hoped that the Manual will prove an attractive introduction to the experimental determination of chemical facts, and will lead the pupil to an interest in chemical theory for its own real and permanent value.

The authors gratefully acknowledge indebtedness to that large body of chemistry teachers whose kind reception of the "First Principles of Chemistry" has encouraged them to publish the present laboratory course.

New York, September, 1908.

PREFACE TO REVISED EDITION

The new edition of this Laboratory Manual is the result of the authors' nine years of experience with the first edition; it also embodies the suggestions of many other chemistry teachers throughout the country.

Such a thorough testing has led to the simplification of some experiments and to the modification of others. A special attempt has been made to have all questions on fact and theory so simple that the answers may reasonably be expected from students of average ability. Whenever additional information is needed to throw light upon the discussion, it has been freely furnished.

A number of new experiments have been added in order to offer a greater range of material, so that selections can be made to fit the aims of a particular course. These additions deal with both the practical and the theoretical sides of the subject.

The typography and the arrangement of the questions have been designed so as to indicate each step in the experiments. Blank spaces for answers to the questions and room for drawings make possible a use of the book as manual and laboratory notebook combined. This plan is growing in favor, since it gives the student more time for experimentation and observation of laboratory phenomena. For the teacher, on the other hand, there is the advantage that the correcting of notebooks may be done with less drudgery and with greater efficiency.

A number of new illustrations have been added to lend interest to the work and to suggest to the students a suitable assembling of the apparatus. Half-tones have been used in preference to line drawings, so that the student will have to depend upon himself in putting into his laboratory notebook the line drawings of the apparatus.

New York, June, 1917.

GENERAL SUGGESTIONS TO TEACHERS

Selection of Experiments. — The time usually allotted to the laboratory work in the first course in Chemistry is not sufficient for performing all the experiments given in this manual. As an aid in the selection of a well-balanced course, the experiments are divided into the following groups:

GROUP A. 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 16, 18, 19, 20, 22, 23, 24, 26, 27, 34, 36, 37, 38 or 39, 41, 42, 43, 44, 46, 47, 50, 51, 53, 61, 62, 63, 66, 67, 68, 71, 78, 79, 80.

GROUP B. 12, 14, 15, 17, 21, 25, 28, 33, 48, 49, 55 or 56, 64, 65.

GROUP C. 13, 29, 30, 31, 32, 35, 40, 45, 52, 54, 57, 58, 59, 60, 69, 70, 72, 73, 74, 75, 76, 77, 81, 82, 83, 84.

It is recommended that all students be required to perform the experiments of Group A. These experiments are fundamental in their nature, and very valuable as a means of laboratory instruction. This list, together with a certain number of experiments from Group B, will satisfy the usual college entrance requirement in Chemistry.

Most instructors will doubtless assign to their classes a good portion of the exercises in Group B. The several quantitative experiments in this subdivision are valuable for their training in manipulation, for the theory they illustrate, and for the interest they arouse.

It is hoped that every laboratory section will find time for some of the experiments in Group C, particularly those dealing with the practical applications of Chemistry.

The Directions for the Experiments. — At first the directions for the laboratory operations are somewhat detailed. This plan has been followed in order that the beginner may have the help needed to perform the experiment readily and intelligently. As the student gains in experience and self-reliance, the directions become less full.

All the questions have been put in italics so that the student will realize their importance. They should be answered in regular order for two reasons: (1) so that the student will understand what he is doing at the time the question is asked; and (2) so that he may have information needed for later parts of the experiment. The authors have taken great care to avoid questions that the student cannot fairly or legitimately answer from the experimental data, and they have not hesitated to give fact or theory when these are necessary to a fair comprehension of the questions. Formulas for products new to the experience of the student have been given in the form of equations to be completed.

When *Class Discussion* appears in parentheses, it means that the student requires further information in order to give a complete answer. Such information is often best furnished in a class discussion.

When a separate notebook is used, the *tabular forms for numerical data* should be written in at the beginning of the experiment, so that the measurements may be recorded as soon as they are made. Some instructors find it advantageous to have the students put in the tabular forms before coming into the laboratory.

Apparatus and Material. — It has been the aim of the authors to use such simple forms of apparatus as are commonly found in the ordinary laboratory equipment. For their general availability, attention is called to the agate pans and the Syracuse form of watch glasses. This watch glass is superior to glass plates for covering and handling bottles of gas. Although the brass capsule, ramrod, and holder used in Experiments 9 and 17 can be purchased, many instructors will prefer to have them made in the laboratory shop. Accordingly, directions for making them are inserted here.

The sodium capsule is made either (*a*) by cutting $\frac{1}{4}''$ brass tubing ($\frac{1}{32}''$ wall) into pieces about an inch long, and soldering into one end a brass disk $\frac{1}{8}''$ thick; or (*b*) by drilling $\frac{5}{16}''$ brass rod with a $\frac{1}{8}''$ or $\frac{3}{16}''$ drill. The latter can be readily done by mounting the rod in a draw-in chuck in a lathe, first drilling and then cutting off.

The handle consists of a piece of No. 14 copper or brass wire. A few turns are wrapped tightly around the capsule, and about 8″ of the wire project at right angles to the capsule. The outer end of

the wire should either be bent into a flat loop or be forced into a short piece of dowel rod.

A ramrod of iron or brass, about 5″ long, sliding easily into the capsule, should be provided.

In the lists of material, *concentrated acid* means acid of the indicated specific gravity: hydrochloric acid, 1.19, sulphuric acid, 1.84, and nitric acid, 1.42. The concentrated ammonia water should have a specific gravity of 0.90.

For *dilute acids* and ammonium hydroxide the authors commonly employ the following concentrations:—

Ammonium hydroxide (1 : 4), that is, one part by volume of concentrated ammonia water to four volumes of water.

Hydrochloric acid (1 : 4)
Nitric acid (1 : 4)
Sulphuric acid (1 : 6)

Early in the course, all students should be given definite directions for the safe mixing of concentrated sulphuric acid with water. The required amount of water should be measured out. Then small portions of the concentrated acid should be poured slowly into the water, and the mixture should be agitated after each addition.

In many cases special concentrations for acids and other solutions are given at the head of the experiment. When no concentration is expressed, one to ten is understood, that is, one part by weight of the chemical to ten parts by weight of water. (A cubic centimeter of water at ordinary temperature is considered to weigh one gram.) In the majority of cases, however, one to twenty solutions will be found to work quite as well as the one to ten, with a consequent saving of reagents.

It will be found convenient to have ready for the students when they come into the laboratory the solutions listed in the *Material* for the various experiments.

Several of the experiments require solid chemicals in small amounts. In such cases, the authors have often found it advisable to distribute the chemicals on labeled slips of paper (about 5 × 10 cm.), arranged in places easily accessible to the students.

In taking specified quantities of solutions, students may need graduates in some cases; but more frequently the necessary quan-

tity may be measured as a fraction of a test-tubeful. The ordinary test-tube ($6 \times \frac{3}{4}''$) contains 30 cc.

In cases when only one or two cubic centimeters of a solution are to be taken, the solutions can be drawn from burettes, which should be labeled and accessible.

CONTENTS

		PAGE
Care and use of apparatus		1
Drawings		3
Laboratory work and its record		5
EXPERIMENT		
1.	Setting up apparatus	7
2.	Heating of metals in air	10
3.	Weight change on heating a metal	12
4.	Decomposition of a compound formed by heating a metal in air	14
5.	Determination of the percentage of oxygen in air	16
6.	Preparation of oxygen	19
7.	Formation of oxides	21
8.	Electrolysis of water	23
9.	Decomposition of water by sodium	25
10.	Preparation of hydrogen	26
11.	Properties of hydrogen	28
12.	Distillation of water	30
13.	Solution and suspension	33
14.	Temperature and relative solubility	35
15.	Water of crystallization	37
16.	Equivalent of magnesium	39
17.	Equivalent of sodium	42
18.	Preparation and properties of chlorine	45
19.	Preparation and properties of hydrochloric acid	48
20.	Test for a chloride	51
21.	Weight of a liter of oxygen	54

EXPERIMENT		PAGE
22.	Bases	57
23.	Alkalies	61
24.	Neutralization	64
25.	Titration	67
26.	Types of chemical change. Direct combination. Simple decomposition	70
27.	Types of chemical change. Simple replacement	73
28.	Types of chemical change. Double decomposition	76
29.	Salts that are not neutral	79
30.	Flame tests	82
31.	Preparation of an acid salt	84
32.	Preparation of sodium carbonate	85
33.	Determination of water of crystallization	86
34.	Forms of sulphur	89
35.	Preparation of metallic sulphides	92
36.	Preparation and properties of hydrogen sulphide	94
37.	Sulphur dioxide, sulphite method	96
38.	Sulphur dioxide, reduction method	99
39.	Properties of sulphuric acid	102
40.	Preparation of nitrogen	106
41.	Preparation and properties of ammonia	109
42.	Ammonium compounds	111
43.	Preparation and properties of nitric acid	115
44.	Preparation and properties of nitric oxide	118
45.	Preparation and properties of nitrous oxide	121
46.	Preparation and properties of bromine	123
47.	Preparation and properties of iodine	126
48.	The halogen acids	129
49.	Destructive distillation	132
50.	Properties of carbon	135
51.	Preparation and properties of carbon dioxide	138

EXPERIMENT PAGE

52. Chemical fire extinguisher 141
53. Hard waters 144
54. Baking powders 148
55. Preparation and properties of carbon monoxide . . . 152
56. Preparation and properties of carbon monoxide, oxalic acid method 154
57. Borax and boric acid 157
58. Water softening 160
59. Bleaching of cotton 163
60. Four ways of preparing a salt, sodium chloride . . . 166
61. Cobalt nitrate tests 170
62. Borax bead tests 172
63. Identification of simple salts 174
64. Action of metals on salt solutions 176
65. Equivalent of silver 179
66. Tests for iron salts 182
67. Action of a reducing agent on a ferric salt 184
68. Action of an oxidizing agent on a ferrous salt . . . 186
69. Iron salts in photography — blue prints 187
70. Silver salts in photography 190
71. Aluminum hydroxide 192
72. Dyeing: substantive, salt, or direct colors 194
73. Dyeing: acid colors 198
74. Dyeing: basic colors 201
75. Double salts 204
76. Qualitative separation of lead, silver, and mercury . . 210
77. Chromium compounds 214
78. Fermentation 216
79. Preparation of ethereal salts 220
80. Soap making 222
81. Starch 223

EXPERIMENT PAGE

82. Food constituents. Fats. Proteins 226
83. Food constituents. Carbohydrates 228
84. Constituents of milk 231

APPENDIX

Physical Constants of the Important Elements . . . 234
Table of Solubilities 236
General Rules for Solubility 237
Volatility of Compounds 237
Weight of a Liter of Common Gases 237
The Metric System 238
Pressure of Water Vapor 240
List of Supplies 241

LABORATORY EXERCISES

IN

CHEMISTRY

LABORATORY EXERCISES IN CHEMISTRY

CARE AND USE OF APPARATUS

THE accompanying picture (Figure 1) gives the student the names of pieces of apparatus with which he is not familiar. These articles should be kept in a clean and orderly condition; good results cannot otherwise be secured.

Figure 1. Laboratory apparatus in common use.

a, test tube rack; *b*, bunsen burner; *c*, mortar and pestle; *d*, watch glass (Syracuse); *e*, thistle tube; *f*, flask; *g*, crucible; *h*, reagent bottles; *j*, evaporating dish; *k*, funnel; *l*, beakers.

The Burner. — The bunsen burner should burn with a clear, blue flame. The ordinary gas flame deposits soot on objects which it touches. The character of the flame is regulated by adjusting the quantity of air that enters the holes at the base of the burner. The flame sometimes "strikes back," that is, begins to burn at the base where the air enters. This means

that too large a proportion of air is entering the tube. Give the rubber tubing a sudden, sharp blow with the edge of the hand. If successful, you will extinguish the flame at the base of the burner and produce a colorless flame at the top of the burner. If not successful, turn off the gas, adjust the movable ring, and relight the burner.

Heating Glassware. — Test tubes may be put directly in the flame; beakers and flasks should be protected by wire gauze or asbestos mat. When glass apparatus contains a liquid, the flame should never extend above the liquid in the vessel. *Never* attempt to heat articles made of thick glass, such as bottles and battery jars, because the poor conductivity of glass causes unequal expansion and breakage.

Heating Porcelain. — Evaporating dishes and crucibles can be heated to very high temperatures. Crucibles can be put directly in the flame, but evaporating dishes should be placed on wire gauze with asbestos center. In both cases the heat should be applied slowly at first.

Setting up Apparatus. — (*a*) Have everything firmly arranged and securely placed.

(*b*) Place the weight of the object directly over the base of the ring-stand.

(*c*) Have the rod of the ring-stand *away* from you, not *toward* you.

(*d*) All glass apparatus should be loosely clamped.

(*e*) See that rubber stoppers fit securely, but use care in pressing them into the necks of thin glass articles.

(*f*) Never try to push a glass tube through the hole in a stopper. Moisten the end of the tube, and work it slowly through the hole, with constant turning.

(*g*) The lower end of a thistle tube should dip under the surface of the liquid in the bottle or flask.

(*h*) The bends in glass tubes should be rounding, not angular. The latter are likely to break and the flow of a gas in them is partly obstructed.

(*i*) Pay a good deal of attention to the 'appearance' of your apparatus. Have vertical lines vertical; horizontal lines horizontal.

(*j*) Keep pieces of clean muslin in your locker. The directions frequently call for *dry* tubes and bottles.

(*k*) Do not lay a stopper from a reagent bottle on the laboratory table. Remove the stopper from the bottle by pressing the top of the stopper between the lower joints of the second and third fingers having the palm of the hand upward. This leaves the thumb and forefinger for grasping the bottle.

(*l*) After pouring from a reagent bottle remove the drop of liquid usually sticking to its lip by touching it to the top of the receiving vessel.

Detailed directions for cutting, bending, and "fire-polishing" glass tubing are given in Experiment 1.

DRAWINGS

The purpose of drawings in the laboratory note book is not to *make a picture* of the apparatus, but to show that the pupil understands how it works. For this reason, and also for the

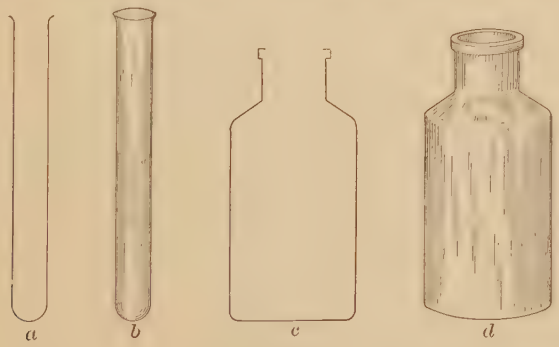


Figure 2.

sake of simplicity, make *sectional*, not perspective drawings. To illustrate this, a test tube and a bottle are shown drawn in

both ways (Figure 2). It will be readily seen that in each case the sectional is the simpler of the two drawings.

In making a sectional drawing, imagine a vertical plane passing through the middle of your apparatus; then imagine your paper to be in the position of this plane. Trace lines where the paper would touch the intersected apparatus. The accompanying diagram (Figure 3) is a sectional drawing of the apparatus used in the preparation of hydrogen.

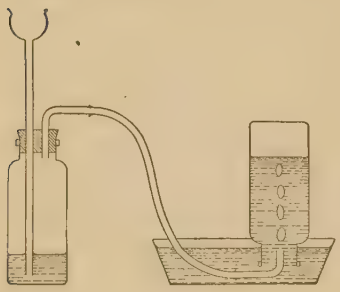


Figure 3.

Notice carefully the following points:

(*a*) The bottom of the pan, in which the gas-collecting bottle stands, is represented by a horizontal straight line; in a perspective drawing this would be a curved line. The bottoms of bottles, flasks, etc., are always represented by straight lines.

(*b*) The rubber stopper is indicated by cross hatching (parallel oblique lines).

(*c*) Water or other liquid is represented by short horizontal lines.

(*d*) A line is not drawn for the top edge of the thistle tube, since this would not show in the imaginary section. A like thing is true for all open bottles and flasks. Thus it is possible to show a passageway for the gases through the apparatus.

The pupil should aim for skill in making these sectional drawings rapidly without the use of a stencil.

LABORATORY WORK AND ITS RECORD

Object. — The purpose of a laboratory is to bring the student in contact with the material studied. This gives a real knowledge of chemical action that book study alone cannot furnish.

See, therefore, all that goes on before you. Then, think about what you see.

Italicized questions in the laboratory directions direct your attention, sometimes to what you should see, sometimes to the meaning of things seen.

Note Book Record. — The record of the laboratory work consists of the making of drawings, the answering of the italicized questions, and the filling in of tabular forms.

In case you use this book for your laboratory record, carefully write all that is called for, using single words or phrases when these will answer the questions. When a longer record is required, form a clear, well-arranged sentence in your mind before setting it down.

In case a separate note book is used for the laboratory record, state *in your own words* what you have done and what you have seen. Avoid copying the laboratory directions, but write simple sentences stating what has been done and its result. This will give a short, clear record.

EXPERIMENT 1

Setting up Apparatus

APPARATUS. Ring-stand, with ring and clamp; flask, 250 cc.; thistle tube; 2-hole rubber stopper to fit flask; piece of glass tubing 23″ long; bunsen burner; wing top for burner; triangular file; asbestos square; pan; rubber connection, 1″ long; wire gauze, asbestos center.

(*a*) Divide the piece of glass tubing into two parts, one about 6 inches long, the other about 17 inches. To do this make a scratch with one forward stroke of a triangular file on one side of the tubing at the point where you desire to cut it. Hold the tube in both hands with the two thumb nails opposite the scratch. Bend the ends of the tubing toward you, at the same time pulling the hands apart (Figure 4). The result should be a clean cut at right angles to the length of the tube.

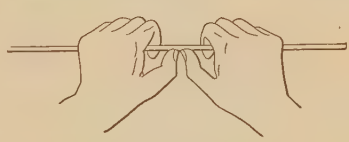


Figure 4.

Place the wing top on the bunsen burner. Light the burner, and turn the ring at the bottom.

What effect has this on the flame?

Adjust the ring so that the flame is blue. Hold the short piece of tubing in the flame so that two inches of the middle portion will be heated. Slowly rotate the tube between your fingers so that all sides are evenly heated. After the heated part has become quite soft, take the tube out of the flame, and, without too much haste, bend it so that the arms make a right angle. See to it that the arms are in the same plane; to do this, sight the tube sideways. Lay the tube aside on the asbestos square to cool. The bend should be rounding and not sharply angular.

Why?

After the tube has become cool enough to hold it at the bend, fire polish it at both ends. To do this hold the end in the upper part of the flame, pointing it downward as much as possible, until the flame turns yellow. This is an indication that the glass has begun to soften.

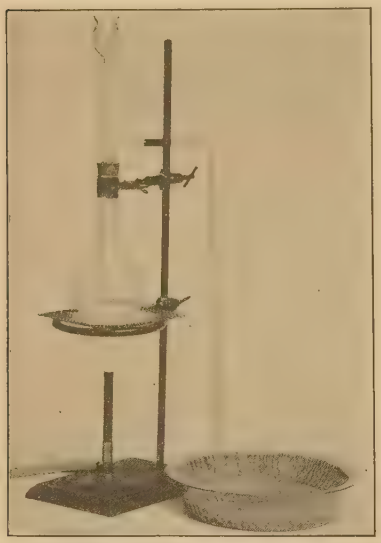


Figure 5.

Bend the long piece of tubing in a similar way, having the bend about three inches from one end. Fire polish both ends.

(*b*) Set up the apparatus shown in Figure 5, paying attention to the following points:

(1) Place the ring-stand with the vertical rod at the back, not the front.

Why?

(2) Have the ring and what it supports directly above the base, not at one side.

Why?

(3) The ring should be placed at such a height that the wire gauze which it supports will strike the flame at the tip of the inner blue cone.

(4) Adjust the clamp so that it holds the flask firmly, but with a very light pressure. Glass used for chemical apparatus is as fragile as other glass.

(5) Moisten the end of the thistle tube and work it slowly,

with constant turning, into one of the holes of the stopper. Never try to push a thistle tube through a stopper.

Why?

(6) Adjust the thistle tube so that its lower end is a quarter of an inch from the bottom of the flask.

(7) Place the short right angle bend in the other hole of the stopper, and connect the long bend by means of a short piece of rubber tubing.

Pour a test tube of water into the flask through the thistle tube. Heat the water till it boils.

Through which tube does the steam issue?

Why should a thistle tube always dip into the liquid in the flask?

Stopper the end of the delivery tube for a moment.

What happens in the thistle tube?

Let the delivery tube dip under the surface of water in a pan. Again boil the water in the flask. Then take away the bunsen flame.

What happens at the lower end of the thistle tube as the steam in the flask cools?

What would happen in this case if the flask carried only a delivery tube without the thistle tube?

If you do not know, repeat the operation, holding the palm of the hand over the upper end of the thistle tube while the contents of the flask are cooling.

Result?

In what two ways does the thistle tube act as a safety device?

EXPERIMENT 2

Heating of Metals in Air

APPARATUS. Bunsen burner; forceps; ring-stand with one ring; pipe-stem triangle; lid of porcelain crucible; iron wire 15 cm. long.

MATERIAL. Copper strips (5 cm. × 1 cm. × 0.5 cm.) or # 24 copper wire; magnesium ribbon, 6 cm.; granulated tin; sandpaper, # 1.

(*a*) **Copper.**

Scour a piece of copper with sandpaper. Examine the bright copper, noting its color, luster, and flexibility. Take hold of one end of the copper with forceps, and hold the other end in the outer flame of the burner until it is red hot. Remove the strip from the flame and watch it while cooling. Bend the strip.

Compare the properties of the surface material with those observed in the original copper and record in the table below.

(*b*) **Magnesium.**

Examine a piece of magnesium ribbon, noting its color, luster, and flexibility. Using forceps, take hold of one end of the magnesium, and place the free end of the ribbon in the flame.

Result ?

Compare the product with the magnesium and record your observations in the table.

(*c*) **Tin.**

Place the lid of a porcelain crucible on a pipe-stem triangle, supported on a ring-stand. On the crucible lid put a few pieces of granulated tin, and heat gently at first, keeping the flame in motion and well below the crucible lid. When the tin melts, stand the burner beneath the crucible lid and stir the tin constantly with an iron wire.

Compare the product with the original tin and record your observations in the table.

TABLE

Material Examined	Color	Luster	Flexibility
Copper			
Substance obtained by heating copper			
Magnesium			
Substance obtained by burning magnesium			
Tin			
Substance obtained by burning tin			

Have chemical or physical changes taken place during the heating of the metals in air?

Explain.

EXPERIMENT 3

Weight Change on Heating a Metal

Each student should perform but one experiment, *a* or *b*.

APPARATUS. (*a*) Porcelain crucible; horn pan balance; shot or sand; pipe-stem triangle; ring-stand; bunsen burner; iron wire.

(*b*) Same as for (*a*), except the iron wire.

MATERIAL. (*a*) Granulated tin; (*b*) copper gauze, or copper wire, #30.

Figure 6.

(*a*) **Tin.**

Counterpoise on a horn pan balance a porcelain crucible that contains about two grams of granulated tin (Figure 6). Remove the crucible, leaving the counterpoise on the balance.

Place the crucible on a pipe-stem triangle. Heat gently at first, keeping the flame in motion and well below the crucible. Gradually increase the heat, and allow the crucible to remain so that it is just above the tip of the inner cone of the flame for twenty minutes. Stir in the tin frequently with an iron wire.

Record any change in appearance.

Remove the burner and allow the crucible to cool on the triangle.

Place the crucible on the balance.

Has there been a loss or a gain in weight?

What explanation can be made of the change in weight?

(Consider the probability of the air having something to do with the change.)

(*b*) **Copper.**

Counterpoise a porcelain crucible, containing about 2 grams of fine copper wire, or fine-meshed copper gauze rolled into a loose ball.

Place the crucible on a pipe-stem triangle. Heat gently at first, keeping the flame in motion and well below the crucible. Gradually increase the heat and then allow the crucible to remain so that it is just above the tip of the inner cone of the flame for thirty minutes.

Remove the burner, put the cover on the crucible, and allow it to cool on the triangle.

Place the crucible without its cover on the balance.

Has there been a loss or a gain in weight?

What explanation can be made of the change in weight?

(Consider the probability of the air having something to do with the change.)

EXPERIMENT 4

Decomposition of a Compound formed by Heating a Metal in Air

APPARATUS. Ring-stand and clamp; hard glass test tube; rubber stopper; delivery tube; dish of water; test tube; splinter; bunsen burner

MATERIAL. Mercuric oxide.

(*a*) Put about 2 grams of the red powder in a hard glass test tube fitted with a stopper carrying a delivery tube. Place

Figure 7.

the end of the delivery tube under the mouth of a test tube filled with water and inverted in a dish of water (Figure 7).

(*b*) Heat *gradually* the hard glass tube.

What is the cause of the bubbling when the tube is first warmed?

Do not allow the hard glass test tube to cool while the mouth of the delivery tube is under water.

Why?

As soon as one half the water in the inverted tube is displaced, remove the test tube, invert it, and insert a glowing splinter.

Result ?

(*c*) Collect a second test tube of gas and test as before.

What is the difference between the behavior of the glowing splinter in the two test tubes?

What was the gas in the first test tube ?

The gas in the second test tube was oxygen.

(*d*) Take a splinter and scrape together the substance which has collected on the cooler portion of the hard glass tube.

What is the substance ?

Of what is the red powder composed?

Complete the equation:

mercuric oxide $\longrightarrow$ _____ + _____

SECTIONAL DRAWING OF APPARATUS

EXPERIMENT 5

Determination of the Percentage of Oxygen in Air (Volumetric)

APPARATUS. Glass cylinder, about 12″ × 2″ (hydrometer jar); 50 cc. gas-measuring tube; # 18 copper wire, 23 cm. long; evaporating dish; thermometer; barometer.

MATERIAL. Yellow phosphorus.

Caution! Yellow phosphorus should never be handled except under water.

(*a*) Nearly fill a glass cylinder with water. Pour into a 50 cc. gas-measuring tube enough water so that the water level is at a graduation mark near the bottom of the tube when it is inverted and its mouth placed under the water in the cylinder. In taking readings the tube should be held between thumb and finger so as not to heat the inclosed gas, and it should be so adjusted that the water is at the same level outside and inside. Have your eye at the level of the water.

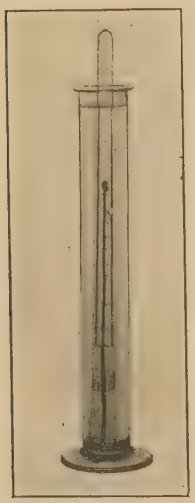


Figure 8.

In a table like that indicated below, record the volume of the inclosed air and its temperature. Get this by taking the temperature of the water, which should have stood long enough to be at the temperature of the room. *Also obtain and record the barometer reading. Applying Charles' law and Boyle's law, find the volume that this air would occupy at standard conditions* (0° *C. and* 760 *mm.*).

Take to the instructor an evaporating dish of water and obtain a wire with a piece of phosphorus on one end. **Keep the phosphorus under water until you reach your desk and immediately put the wire with the phosphorus into the cylinder of water,** letting it rest against the side of the cylinder (Figure 8). Now lower the gas-measuring tube containing the measured volume of air over the phosphorus so that the phosphorus will be above the middle of the space occupied by the air.

Note and record the action of the phosphorus.

Is there any change in the level of the water inside the tube?

The substance formed is phosphorus oxide, which dissolves in the water.

Complete the equation:

_____ + _____ ⟶ phosphorus oxide

Allow the action to continue overnight.

(*b*) The next day, remove the tube from over the phosphorus, but still keep the mouth of the tube beneath the water. Then raise or lower the tube until the water is at the same level inside and outside.

Read and record in the table the volume of the gas remaining in the tube. Record the temperature of the water and the barometric pressure.

Applying Charles' law and Boyle's law, find the volume which the gas that remains would occupy if it were at standard conditions. The difference between the original volume of air inclosed and the volume of gas that remains, both corrected to standard conditions, represents the amount of oxygen removed by the phosphorus. *Calculate the percentage of oxygen in the air.* Make all calculations in your note book.

TABLE

Volume of air taken	cc.
Temperature of air taken	°C.
Pressure of air barometer reading	mm.
Corrected volume of air	cc.
Volume of gas remaining in measuring tube	cc.
Temperature of gas remaining	°C.
Pressure of gas remaining (barometer reading)	mm.
Corrected volume of gas remaining	cc.
Volume of oxygen removed by phosphorus	cc.
Percentage by volume of oxygen in air	%

CALCULATIONS

EXPERIMENT 6

Preparation of Oxygen by the Decomposition of a Chlorate

APPARATUS. Two test tubes; delivery tube; rubber stopper; ring-stand with clamp; bunsen burner; four 6-oz. wide-mouth bottles; four glass plates for wide-mouth bottles; enameled pan, or pneumatic trough; watch glass; funnel.

MATERIAL. Potassium or sodium chlorate; manganese dioxide; filter paper; splinter.

In this experiment there is danger of the water "sucking back" into the hot test tube. Guard against this by removing the delivery tube from the water before the flow of gas stops.

(*a*) Mix thoroughly about 8 grams of potassium chlorate and 6 grams of manganese dioxide. Place in a test tube provided with a delivery tube. Clamp the test tube in a position

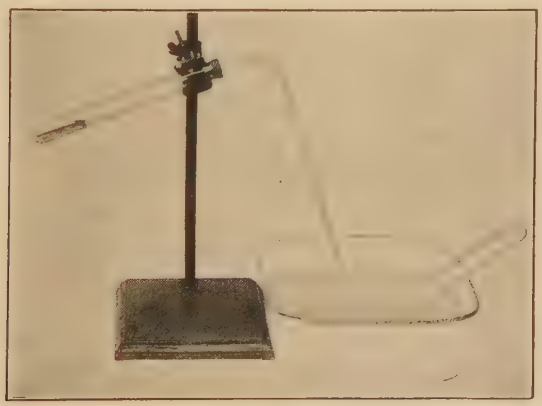


Figure 9.

convenient for heating (Figure 9). If a pneumatic trough is used, adjust the delivery tube to deliver the gas just below the opening in the bridge of the trough. Carefully regulate the heating so as to cause a very gentle evolution of the gas. **Do not heat the test tube sufficiently to make the flame yellow.**

(*b*) Collect a test-tubeful of the gas and test it with a glowing splinter.

Result ?

Collect the remainder of the gas in wide-mouth bottles. Cover with glass tubes and keep for Experiment 7.

(*c*) Remove the delivery tube and allow the test tube to cool. Nearly fill the test tube with hot water, close the mouth of the tube with the thumb, and shake the tube.

Pour the muddy liquid on a moistened filter paper fitted to a funnel. Collect the clear liquid (*filtrate*) in a test tube.

Remove a small portion of the black residue from the filter, place it on a second watch glass, and set it aside to dry.

Which of the original substances does the black residue resemble ?

Taste a crystal of potassium chlorate. Then taste the clear filtrate.

Do they taste alike?

Is the substance in the filtrate potassium chlorate or a different substance ?

From which of the original substances was the oxygen probably derived ?

Complete the equation :

potassium chlorate ⟶ potassium chloride + _____

{ potassium, chlorine, oxygen } { potassium, chlorine }

The manganese dioxide causes the oxygen to be liberated more regularly and at a lower temperature.

EXPERIMENT 7

Formation of Oxides

APPARATUS. Deflagrating spoon; asbestos paper; bunsen burner.

MATERIAL. Four bottles of oxygen; splinter of charcoal; magnesium ribbon, 5 cm. long; sulphur; red phosphorus.

(*a*) Place a thin splinter of charcoal across the bowl of a clean deflagrating spoon. Heat the end of the splinter until it glows brightly, and immediately lower it into a bottle of oxygen.

Does the charcoal burn with a flame?

Compare the intensity of the action in oxygen with that in air.

Of what elements does the gas formed probably consist?

(*b*) Twine a piece of magnesium ribbon around the rod of the deflagrating spoon, allowing the upper end to project slightly. Light the free end and lower the spoon into a bottle of oxygen.

Compare the combustion of the magnesium with that of the carbon.

Compare the action in oxygen with that in air.

What is the appearance of the oxide of magnesium?

Does this seem to be the same material as that obtained when magnesium was burned in air (Experiment 2)?

(*c*) Clean the spoon, line it with asbestos paper (baking sheet), and put on the paper a small piece of sulphur. Heat the sulphur by directing the flame of the burner against it until it lights, and then lower it into the bottle of oxygen.

Describe the burning of the sulphur in oxygen.

When the mist (principally unburned sulphur) has disappeared, *very cautiously* smell the contents of the bottle.

Name this gas.

(*d*) Reline the spoon, and place on the asbestos a pinch of red phosphorus. Heat the phosphorus until it lights, and put it into a bottle of oxygen.

Describe the burning of phosphorus in oxygen.

Is the product a gas or does it consist of fine solid particles?

Name the product.

Make a general statement as to the relative rapidity of the burning of a substance in oxygen and its burning in air.

Give a general name for the product formed by burning an element in oxygen.

Complete the equations:

carbon + oxygen ⟶ _____

magnesium + oxygen ⟶ _____

sulphur + oxygen ⟶ _____

phosphorus + oxygen ⟶ _____

EXPERIMENT 8

Electrolysis of Water

APPARATUS. Electrolysis apparatus like that shown in Figure 10; battery jar, 4 × 5″; 3 dry cells and connections, or 100-watt lamp and socket with connections for 110 volt direct current; bunsen burner; two test tubes.

MATERIAL. Sulphuric acid, 1 to 20; splinter.

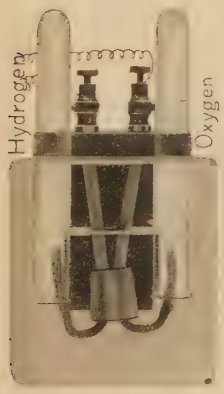


Figure 10.

(*a*) In a small battery jar, place water containing 1 part of sulphuric acid to 20 of water. The use of the acid is to make possible the passage of the current through the water.

Set the electrolysis block firmly on one side of the jar (Figure 10). Fill two test tubes with a mixture of acid and water, cover the end of each tube in turn with your forefinger, and invert it into the water of the battery jar. Remove the finger after the mouth of the test tube is below the surface of the water. Slip each tube into the clamp on one side of the small board and carefully push it down over the electrode until the latter is entirely within the tube. Thoroughly rinse the hands to remove all traces of acid.

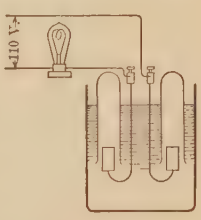


Figure 11.

(*b*) Connect the two binding posts with a battery of three dry cells in series, or with a 110-volt direct current circuit, having a 100-watt incandescent lamp in series between the source of current and the electrolysis apparatus (Figure 11). Determine the direction of the current as directed by the instructor. The electrode through which the current enters is the *anode* (+). The current leaves the solution at the *cathode* (−).

As soon as the water in one of the tubes has been displaced by gas, remove the tube from the battery jar, keeping it mouth downward. Hold the mouth of the tube to a flame.

Result ?

When the other tube has filled with gas, close it with the thumb and remove it from the battery jar, inverting it at the same time. Insert a glowing splinter into the gas.

Result ?

What gas collects at the anode ?

Is the same gas liberated at the cathode ?

(*c*) Refill the tubes with acidulated water and again place them over the electrodes.

How does the amount of gas liberated at the anode compare with the amount liberated at the cathode ?

There is the same amount of sulphuric acid at the end of the experiment as at the beginning.

Where did the gases come from ?

Complete the equation:

water $\longrightarrow$ _____ + _____

EXPERIMENT 9

Decomposition of Water by Sodium

APPARATUS. Brass capsule, provided with a holder; brass ramrod to fit capsule; pan; test tube or bottle; bunsen burner.

MATERIAL. Sodium.

Caution! The action of sodium with water is very violent. Avoid danger by following directions.

Nearly fill a metallic capsule with freshly cut sodium from which *all* the crust has been removed. **The sodium must be pressed firmly into the capsule.**

Figure 12.

Place the capsule in a wire holder, and, holding the capsule mouth *downward*, thrust it under the mouth of an inverted test tube or small wide-mouth bottle filled with water. Control the evolution of the gas by *slightly* inclining the capsule (Figure 12). If careless handling allows the sodium to escape from the capsule, *stand aside.*

When the test tube is filled with gas, carry it mouth downward to a flame.

Result?

This gas is hydrogen. The yellow color of the flame is due to sodium. Sodium is an element.

Where does the hydrogen come from?

Complete the equation:

$$\text{sodium} + \text{water} \longrightarrow \underline{\qquad\qquad} + \text{sodium hydroxide}$$

$$\left\{\begin{matrix}\text{hydrogen}\\ \\ \text{oxygen}\end{matrix}\right. \qquad\qquad \left\{\begin{matrix}\text{sodium}\\ \text{hydrogen}\\ \text{oxygen}\end{matrix}\right.$$

EXPERIMENT 10

Preparation of Hydrogen by the Reaction between an Acid and a Metal

APPARATUS. Wide-mouth bottle, 8 oz.; two-hole stopper; thistle tube; delivery tube; pneumatic trough; three wide-mouth bottles, 6 oz.; three glass plates; watch glass; beaker, 250 cc.; test tubes; ring-stand with large ring; bunsen burner.

MATERIAL. Zinc; dilute sulphuric acid, 1 to 6; copper sulphate solution.

Caution! A mixture of hydrogen and oxygen (or air) is dangerously explosive. Have no light or burner near your generator. Collect the gas in test tubes until you find that the gas burns quietly when a flame is applied to it.

(*a*) Use a wide-mouth bottle provided with a two-hole stopper carrying a thistle tube and a delivery tube (Figure 13).

Why must the end of the thistle tube dip below the surface of the liquid?

Have three bottles filled with water, standing inverted in the pneumatic trough. Put about 20 grams of granulated zinc into the generator, and pour through the thistle tube dilute sulphuric acid (1 to 6), until one fourth of the bottle is filled.

Figure 13.

Result?

If the action is slow in starting, add a few drops of copper sulphate solution through the thistle tube.

Observe and describe the action in the generator.

Under no circumstances add more acid, nor in any way interfere with the generator without consulting the instructor.

(*b*) Collect the gas in a test tube. As soon as the test tube is filled, hold it mouth downward to a small flame. Continue to collect and test the gas in this manner until a portion burns quietly. The hydrogen is now ready to be collected for Experiment 11.

Fill three bottles with the gas. Leave them standing on the shelf of the pneumatic trough, or cover them with glass plates and set them mouth downward on the desk.

☞ Proceed to Experiment 11, part (*a*).

(*c*) Filter a few drops of the liquid in the generating bottle into a watch glass. Place this on the top of a beaker one third full of water, and boil the water until a solid has appeared in the watch glass.

Examine this solid and describe its appearance.

The compound is zinc sulphate, which is composed of zinc, sulphur, and oxygen.

Complete the equation:

zinc + sulphuric acid ⟶ _____ + _____

{ hydrogen
sulphur
oxygen }

From what material does the hydrogen come?

Why is the action called a replacement action?

Why was the gas collected in test tubes only and repeatedly tested in the first part of the experiment?

EXPERIMENT 11

Properties of Hydrogen

APPARATUS. Glass tube (20 cm.); test tube; clamp; bunsen burner.
MATERIAL. Copper oxide (wire form); taper.

(*a*) Replace the end of the delivery tube of the generator used in Experiment 10 with a straight, dry glass tube, and let it lead to the bottom of a nearly horizontal test tube that contains a little copper oxide (wire form) (Figure 14). Allow the hydrogen to pass into the tube for two minutes to expel the air. Then heat the tube directly under the copper oxide.

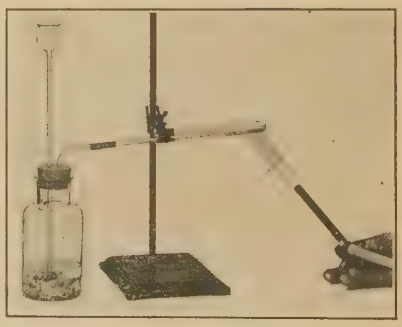


Figure 14.

When the hydrogen passes over the heated copper oxide, what collects in the cool portion of the tube?

What is left in the heated portion?

How do you account for the production of these substances?

What element was removed from the copper oxide by the hydrogen?

A material which acts in this way is called a *reducing agent.*

Complete the equation:

copper oxide + hydrogen ⟶ _____ + _____

{copper, oxygen} { }

(*b*) Holding a bottle of hydrogen mouth downward, thrust into it a lighted taper.

What happens to the flame of the taper?

What is occurring at the mouth of the bottle?

Slowly withdraw the taper from the bottle.

Explain the result.

(*c*) Lift a bottle of hydrogen and hold it, uncovered and mouth downward, for a full minute by the watch. Then hold the mouth of the bottle to a flame.

Result?

Hold another bottle, uncovered and mouth upward, for a full minute, and again hold the mouth to the flame.

Result?

Is hydrogen heavier or lighter than air? Give reasons for your answer.

☞ Now return to Experiment 10, part (*c*).

DRAWING, PART (*a*)

EXPERIMENT 12

Distillation of Water

APPARATUS. Distilling flask, 250 cc., condenser, and tubes as shown in Figure 15; ring-stand; tripod; one ring; clamp; wire gauze; bunsen burner; additional flask for distillation; two beakers, 150 cc., or Erlenmeyer flasks; two small beakers or bottles; watch glass.

MATERIAL. Copper sulphate crystals; solution of phenolphthalein; ammonia water (concentrated).

Arrange the apparatus as shown in Figure 15. Connect one of the rubber tubes to the water faucet so that water will enter

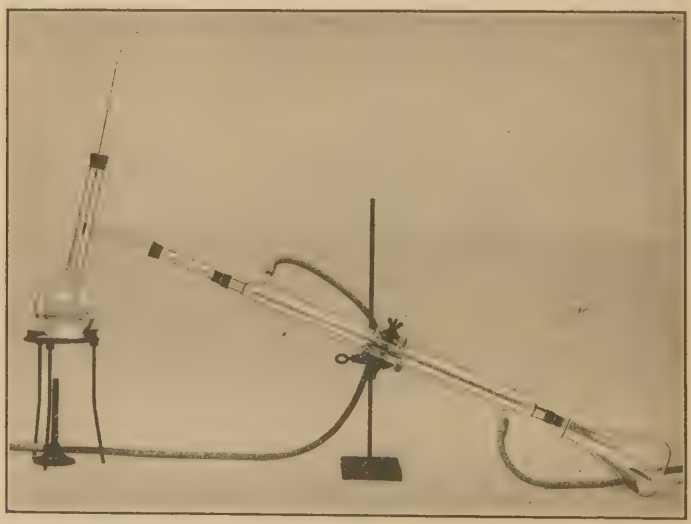


Figure 15.

the lower end of the condenser; let the other drain into the sink.

(*a*) Put into the distilling flask a few crystals of copper sulphate and add 100 cc. of water. Heat the contents of the flask to boiling.

Does the copper sulphate dissolve?

Give a reason for your answer.

Let the distillate (the condensed steam) collect in a beaker or flask.

Does it contain any copper sulphate ?
Give a reason for your answer.

Dissolved solids in general act in distillation as the copper sulphate does.

A substance which readily changes into a gas on being heated is said to be volatile.

Is copper sulphate volatile under the conditions of this experiment ?

How do you know?

Can water be purified from non-volatile impurities by distillation?

(*b*) Add a drop of phenolphthalein solution to some pure water.

Result ?

To 100 cc. of pure water add one drop of concentrated ammonia water and a drop of phenolphthalein solution.

Result ?

This test is used in this experiment to indicate the presence of ammonia in water.

Prepare another mixture of one drop of concentrated ammonia water in 200 cc. of pure water. Pour it into a clean flask and distil. Collect a little of the distillate in a clean beaker or small bottle. Add a drop of phenolphthalein solution.

Does the distillate contain ammonia ?

Is ammonia volatile ?

Continue the distillation for two or three minutes and then collect a second portion of the distillate.

Does it contain any ammonia ?

Does the first portion of the distillate contain more or less ammonia than the second portion ?

Can water be readily purified by distillation from a volatile impurity like ammonia ?

(*c*) Determine, by evaporation on a watch glass over steam, whether water from the faucet contains non-volatile impurities.

Result?

Suggest a method by which salt water can be converted into fresh water.

DRAWING

EXPERIMENT 13

Solution and Suspension

APPARATUS. Beaker, 250 cc.; small battery jar; teaspoon or horn spatula; two test tubes; stirring rod.

MATERIAL. Rock salt; fine salt; calcite; powdered sodium chromate; fine clay or precipitated chalk.

(*a*) Soluble and insoluble substances.

Put a piece of rock salt about the size of a pea into a test tube of water. In another test tube of water put a lump of calcite of equal size. Close the mouth of each of the test tubes and shake them.

Which substance disappears in the water?

Which substance has not dissolved?

How can you tell this from the shape of the pieces of solid remaining in the water?

Name the soluble substance.

(*b*) Distribution of the solute in the solvent.

Put half a teaspoonful of table salt in a beaker of water and stir the mixture.

Is the solution clear and transparent?

Dissolve a teaspoonful of powdered sodium chromate in a beaker of water by stirring thoroughly.

Is the solution clear?

Is it transparent?

What does the uniformity of color indicate concerning the distribution of the dissolved substance (the solute) in the water (the solvent)?

Allow the solution to stand for a short time.

Does the sodium chromate settle ?

Empty the beaker into the receptacle designated by the instructor.

(*c*) **Suspension.**

In a battery jar of water put a teaspoonful of fine clay, or precipitated chalk. Stir thoroughly.

Such a mixture is called a *suspension.*

Allow the jar to stand for a time.

What does the solid in suspension tend to do ?

What differences do you note between a suspension and a solution ?

State three characteristics of a true solution that have been brought out by this experiment.

EXPERIMENT 14

Temperature and Relative Solubility

APPARATUS. Four test tubes; four small rubber bands; bunsen burner.

MATERIAL. Powdered potassium nitrate or powdered magnesium sulphate (Epsom salts); fine salt.

(*a*) Fill a test tube with powdered potassium nitrate (saltpeter), or powdered Epsom salts. Add the potassium nitrate, a very little at a time, to another test tube one-fourth full of cold water, shaking the test tube after each addition. Continue to add the potassium nitrate until a very little of the solid remains after thorough shaking.

Adjust a rubber band at the upper level of the nitrate remaining in the supply tube.

Since the cold water has dissolved all that it can of the potassium nitrate, it is said to be saturated with respect to that substance at the existing temperature.

Heat in the bunsen flame the cold saturated solution of potassium nitrate just prepared, and then add more nitrate from the supply tube. Shake the tube after each addition as before, and dissolve in the hot water as much as you can of the nitrate. During the process keep the liquid hot by heating occasionally in the flame, but take care not to boil off any of the water. As soon as the hot water is saturated with nitrate, set the solution aside to cool for later examination. Mark, with a *second* rubber band, the upper level of the nitrate remaining in the supply tube.

How is the amount of the potassium nitrate that can be dissolved affected by increasing the temperature of the water?

Make a drawing showing the relative positions of the rubber bands on the supply tube. Indicate the amount of the nitrate dissolved respectively in equal volumes of cold and hot water.

(*b*) Examine the solution set aside to cool.

Describe what has happened.

Is the amount of dissolved substance greater or less at the lower temperature?

When the liquid comes to the temperature of the room, is it a saturated or an unsaturated solution?

How can you determine this?

Empty the tubes containing potassium nitrate into the receptacles designed by the instructor.

(*c*) Repeat part (*a*), using fine salt (sodium chloride) instead of potassium nitrate.

Which is increased the more by increase in temperature, the solubility of sodium chloride or that of potassium nitrate?

Make a statement concerning the relative solubility of salt in hot and in cold water.

DRAWINGS

EXPERIMENT 15

Water of Crystallization

APPARATUS. Horn pan balance; two watch glasses, 3 in.; counterpoise; eight test tubes; test tube rack; bunsen burner.

MATERIAL. Crystallized sodium sulphate; copper sulphate; potassium chlorate; zinc sulphate; barium chloride; potassium sulphate; potassium nitrate; alum.

(*a*) Place a small watch glass on the pan of a balance and then add enough crystallized sodium sulphate to counterpoise the weight placed by the instructor in the other pan. Set the crystallized sodium sulphate aside for a half hour.

Then note and record any changes in its appearance.

Again place the watch glass and its contents on the balance.

What does the change in weight indicate?

(*b*) Heat a few crystals of sodium sulphate carefully in a dry test tube, *holding the tube in a horizontal position.*

What collects on the walls of the test tube?

To what is the change in weight noticed in part (a) probably due?

(*c*) In another test tube, gently heat a crystal of blue vitriol (copper sulphate) over a small flame until a white substance is produced.

What important effects have been produced as to color, form, and composition?

After cooling, dissolve the residue in the bottom of the tube in *a few drops* of hot water. Pour the solution on a watch

glass and allow it to stand. Compare the final product with the original substance.

Result?

What took place when the copper sulphate was heated?

What happened when the residue was dissolved and allowed to cool?

(*d*) In separate dry test tubes gently warm a few crystals of potassium chlorate, zinc sulphate, barium chloride, potassium sulphate, potassium nitrate, and alum.

Record the results in the following tabular form:

TABLE

Substance Heated	Is Water Deposited in Cool Portion of Tube?	Appearance of Residue

Do all crystalline substances contain water of crystallization? Illustrate.

EXPERIMENT 16

Equivalent of Magnesium

APPARATUS. Gas-measuring tube, 50 cc.; battery jar; thermometer; barometer.

MATERIAL. Magnesium; concentrated hydrochloric acid; thread; water for the battery jar that has stood long enough to come to the room temperature.

The data for this experiment should be tabulated as shown on page 41.

Pour about 5 cc. of concentrated hydrochloric acid into a gas-measuring tube. Fill the remainder of the tube with water, taking care not to mix the acid and water; the heavier acid will remain at the bottom.

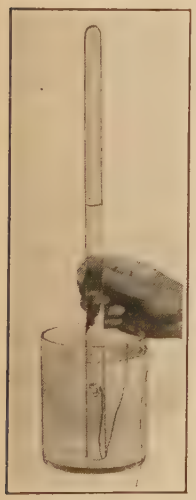


Figure 16.

Roll a piece of magnesium whose exact weight is known (about .045 gram),[1] into a loose coil somewhat smaller than the inside diameter of the tube. Pass a thread through the loop of the coil, and tie it.

Put the magnesium into the measuring tube, holding the thread so that the magnesium will not sink. Close the tube with the thumb, and invert it into a battery jar of water, resting the mouth of the tube against the bottom, so that the thread will be held. Allow the magnesium to rise not quite to the graduation in the tube (Figure 16).

The heavier acid will flow down and react with the magnesium.

What is the gas that collects?

From what substance does it come?

[1] Weigh a strip of magnesium ribbon several meters in length. By calculation, determine the length of a piece that will weigh the required amount. Cut off pieces of this length.

Complete the equation:

magnesium + hydrochloric acid ⟶ { hydrogen / chlorine

_____ + _____ { _____

Why is this reaction termed a replacement?

When the action has ceased (all the metal being dissolved), adjust the levels.

Read the volume of hydrogen obtained and record your result.

Record the temperature of the liquid in the jar and the barometric pressure. Correct the pressure for aqueous tension. (*Use the table on page* 240.) *Make all calculations on page 41.*

Reduce the volume of hydrogen to standard conditions.

The weight of 1000 cc. (1 liter) of hydrogen is 0.09 g. *Calculate the weight at standard conditions of the hydrogen that was produced by the reaction, using the proportion:*

$$1000 \text{ cc.} : \frac{\text{corrected volume}}{\text{of hydrogen}} :: 0.09 \text{ g.} : x \text{ g.}$$

This result is the weight of hydrogen that is liberated by the action of your known weight of magnesium.

Calculate how much magnesium would have been necessary to liberate 1 gram of hydrogen.

This weight is called the equivalent of magnesium.

The equivalent of any element is the weight of that element that replaces (or combines with) 1 gram of hydrogen.

TABLE

Weight of magnesium taken	g.
Volume of hydrogen obtained	cc.
Temperature	° C.
Barometric pressure	mm.
Aqueous tension	mm.
Corrected pressure	mm.
Volume of hydrogen under standard conditions	cc.
Weight of hydrogen (calculated)	g.
Equivalent of magnesium (calculated)	

CALCULATIONS

EXPERIMENT 17

Equivalent of Sodium

APPARATUS. Metallic capsule and holder; brass ramrod to fit capsule; horn pan balance; weights; 16 oz. bottle; graduate, 500 cc.; pneumatic trough; glass plate; barometer; thermometer.

MATERIAL. Sodium.

Caution! Remember that the action of sodium with water is very violent.

The data for this experiment should be tabulated as indicated on page 43.

(*a*) Weigh a metallic capsule that is clean and dry.

Record its weight in the table.

Nearly fill the capsule, as in Experiment 9, with freshly cut sodium freed from any adhering crust. Wipe off any oil with filter paper. Weigh the capsule and contents quickly.

Record the weight.

(*b*) Fill the wide-mouth bottle with water and measure its capacity by pouring the water into a graduate.

Record the volume.

(*c*) Then fill the bottle with water and invert it in the trough. Place the metallic capsule in its holder. Raise the bottle in the trough with the left hand. Take the holder in the right hand and incline it so that the *open end* of the capsule will be *downward*. *Keeping the open end downward*, thrust the capsule under the mouth of the bottle. Control the evolution of the hydrogen by *slightly* inclining the capsule.

(*d*) When the action ceases, adjust the bottle so that the liquid on the inside is level with that outside. Close the mouth of the bottle with a glass plate, remove it from the trough, and set it on the desk, mouth upward. Pour the liquid now in the bottle into a graduate.

Record its volume.

Record the temperature of the liquid in the trough and the barometric pressure. Correct the pressure for aqueous tension. (*Use table on page* 240.)

Reduce the volume of hydrogen to standard conditions. The weight of one liter (1000 cc.) of hydrogen at standard conditions is 0.09 gram.

Calculate the weight of hydrogen evolved. Then determine what weight of sodium is required to liberate 1 gram of hydrogen.

This weight is called the equivalent of sodium.

The equivalent of any element is the weight of that element that replaces (or combines with) 1 gram of hydrogen.

Make all calculations on page 44.

TABLE

Weight of capsule and sodium	g.
Weight of capsule	g.
Weight of sodium taken	g.
Capacity of bottle	cc.
Volume of liquid left in the bottle	cc.
Volume of hydrogen obtained	cc.
Temperature of hydrogen	° C.
Barometric pressure	mm.
Aqueous tension	mm.
Corrected pressure	mm.
Volume of hydrogen at standard conditions	cc.
Weight of the hydrogen (calculated)	g.
Equivalent of sodium (calculated)	

CALCULATIONS

EXPERIMENT 18

Preparation and Properties of Chlorine

APPARATUS. Flask; two-hole rubber stopper; thistle tube; four wide-mouth bottles (6 oz.), with two-hole rubber stoppers; eight glass bends; four rubber connectors; ring-stand with large ring; pan of water; bunsen burner; four glass plates or watch glasses (Syracuse form); one hydrogen generator, provided with jet tube, for the class.

MATERIAL. Concentrated hydrochloric acid; manganese dioxide; taper; colored cloth; powdered antimony.

Caution! Chlorine is a poisonous gas. Do not inhale it. Inhaling ammonia or alcohol will counteract some of its effects.

(*a*) Preparation.

Arrange apparatus as in Figure 17. Pour a small amount of water into the fourth bottle. If good hoods are available,

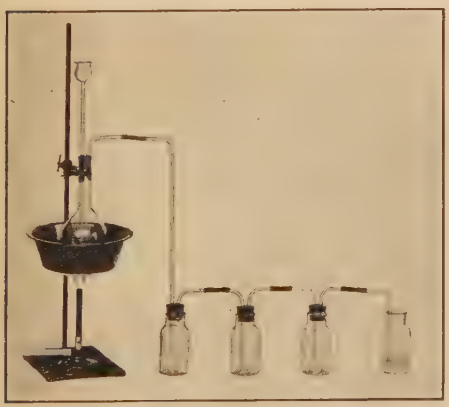


Figure 17.

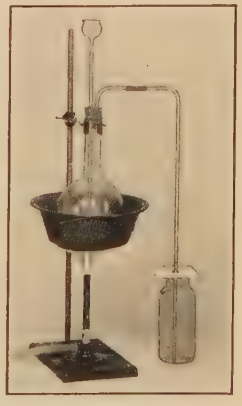


Figure 18.

use the apparatus shown in Figure 18. In this case, the solubility should be determined by passing the gas into a test tube of water.

Place in the flask 15 cc. of concentrated hydrochloric acid and

add about 8 grams of granular manganese dioxide. Rotate the flask so as to mix its contents, then replace the stopper.

Heat the water in the dish under the flask to boiling.

Describe the action in the generator.

(*b*) **Physical properties.**

Hold a piece of white paper behind the first bottle.

What is the color of chlorine?

How can you tell when the bottle is filled with gas?

When the bottles are filled with chlorine, withdraw the flame. Keep the gas for use in parts (*c*), (*d*), and (*e*).

Is chlorine soluble in water?

Give a reason for your answer.

(*c*) **Chemical properties.**

The instructor at this point should introduce a jet of burning hydrogen into a bottle of chlorine.

What compound is formed when hydrogen burns in oxygen?

What is formed when hydrogen burns in chlorine?

The instructor should also sprinkle a pinch of powdered antimony into a bottle of chlorine.

Result?

Is oxygen necessary for combustion?

Lower a lighted taper into a bottle of chlorine. The taper is composed of compounds containing hydrogen and carbon.

Which of these elements is liberated when the candle burns in chlorine?

(*d*) **Bleaching action of chlorine.**

Place a piece of dry colored cloth in one bottle of chlorine and a piece of wet cloth in another. Cover the mouths of the bottles with glass plates.

What must be the condition of the cloth to be bleached by chlorine?

Explain the bleaching of cloth by chlorine. (Class discussion.)

(*e*) **General questions.**

Hydrochloric acid is a compound of hydrogen and chlorine.

With what does the oxygen of the manganese dioxide combine?

What is your conclusion as to the chemical activity of chlorine as compared with oxygen.

With what elements does chlorine readily combine?

DRAWING

EXPERIMENT 19

Preparation and Properties of Hydrochloric Acid

APPARATUS. Flask, 250 cc., with stopper carrying thistle tube and delivery tube; ring-stand with one ring and one clamp; wire gauze with asbestos center; bunsen burner; two test tubes; wide-mouth bottle; enameled pan.

MATERIAL. Sodium chloride; sulphuric acid, 2 to 1; blue litmus paper; magnesium; zinc.

(*a*) Preparation.

Pour about 20 cc. of sulphuric acid (2 to 1) into a flask supported on a wire gauze on a ring-stand, and add about 10 grams of sodium chloride. Gently rotate the flask so as to mix the acid with the chloride. Close the flask with a stopper carrying a thistle tube and a delivery tube arranged for the collection of gas in a dry test tube by downward displacement. If necessary, heat the flask with a *small* flame.

Describe the action in the generator.

Of what elements is hydrogen chloride composed?

Which of the original materials furnished the chlorine?

Which furnished the hydrogen?

In this particular case, sulphuric acid is used because it does not vaporize below 338°, so that none of it passes off with the hydrogen chloride.

sodium chloride	+	sulphuric acid	→	sodium sulphate	+	hydrogen chloride
sodium, chlorine		hydrogen, sulphur, oxygen		sodium, sulphur, oxygen		_____, _____

Why is hydrogen chloride collected by the method used?

(*b*) **Solubility of hydrogen chloride.**

Fill a dish with water and set it on the table. Take the test tube of gas collected by the downward displacement of air, close its mouth tightly with the thumb, invert the test tube, and hold its mouth below the surface of the water. Remove the thumb.

Result?

Explain why the gas is not collected over water.

Close the mouth of the test tube with the thumb and remove it from the water. Moisten a piece of litmus paper with the liquid contained in the test tube.

Result?

Taste the liquid.

Result?

These effects are typical of the water solution of acids. The solution in the reagent bottle marked "hydrochloric acid" is also prepared by dissolving hydrogen chloride in water.

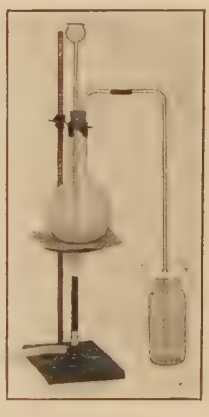


Figure 19.

(*c*) **Density of hydrochloric acid.**

Pour not more than 10 cc. of water into a wide-mouth bottle. Place the mouth of the delivery tube within half a centimeter of, *but not touching*, the surface of the water in the bottle (Figure 19). Heat the flask with a small flame, or by a pan of boiling water, for at least ten minutes. While doing this, occasionally look through the water in the bottle horizontally.

Is the solution of hydrogen chloride formed heavier or lighter than water? Explain.

(*d*) **Action of hydrochloric acid with metals.**

Pour half of the solution just made into a test tube and drop into it a strip of magnesium. Bring a flame near the mouth of the tube.

Results?

Place a piece of zinc in another test tube and pour the remaining hydrochloric acid upon it. Test the gas with a flame.

Results?

What substance is liberated when hydrochloric acid reacts with these metals?

Magnesium and zinc are elements.

When hydrochloric acid reacts with these metals, where does the substance that is liberated come from?

What becomes of the metal?

What three properties have been mentioned as characteristic of acids?

DRAWING

EXPERIMENT 20

Test for a Chloride

APPARATUS. Six test tubes; test tube rack.

MATERIAL. Dilute hydrochloric acid; dilute nitric acid; solutions of silver nitrate, ammonium hydroxide, sodium phosphate, and potassium oxalate; unknowns.

(*a*) To a little hydrochloric acid in a test tube add silver nitrate solution. The substance which separates out is silver chloride.

Complete the equation:

hydrochloric acid + silver nitrate ⟶

_____ + _____

Any solid which thus separates out of a clear liquid is known as a precipitate.

Describe the silver chloride precipitate as to color and appearance.

Set aside the tube containing the precipitate.

(*b*) Take a little of a solution of sodium phosphate and add to it silver nitrate solution.

Result?

Complete the equation:

sodium phosphate + silver nitrate ⟶

silver phosphate + _____

Describe the color and appearance of the silver phosphate.

Set the tube aside.

(*c*) Take a little of a solution of potassium oxalate and add to it silver nitrate solution.

Result?

Complete the equation:

potassium oxalate + silver nitrate ⟶
silver oxalate + _____

Describe the color and appearance of the precipitate.

Set the tube aside.

(*d*) Add nitric acid to each of the three tubes containing the three precipitates.

What happens in each case?

How can you distinguish silver chloride from silver phosphate and form silver oxalate?

How can silver chloride be obtained from any soluble chloride?

What two steps would be necessary to distinguish any soluble chloride from any soluble phosphate or oxalate?

This procedure serves as a means of distinguishing chlorides from other salts.

Describe a test for a chloride.

(*e*) Make another portion of silver chloride. Let the precipitate settle, drain off most of the clear liquid, and add ammonium hydroxide.

Result ?

(*f*) Make still another portion of silver chloride and see what effect light has on it. If possible, stand the tube in the direct sunlight.

What further characteristics of silver chloride are shown in parts (e) and (f) ?

(*g*) Apply to the instructor for an "unknown." Test it for a chloride, writing the results obtained in each step.

State your opinion as to whether the unknown contained a chloride, and give reasons for your belief.

TABLE

Number of Unknown	Effect of Adding Silver Nitrate	If Precipitate is Obtained, Effect of Nitric Acid on It

EXPERIMENT 21

Weight of a Liter of Oxygen

APPARATUS. Horn pan balance; weights; test tube with rubber stopper carrying a delivery tube; 4 in. U-tube with one-hole rubber stoppers and delivery tube; pneumatic trough; 2-liter bottle (acid bottle); glass plate; graduate; ring-stand; clamp; bunsen burner.

MATERIAL. Well dried potassium chlorate; dry, powdered manganese dioxide (C. P. quality gives a more accurate result); granulated calcium chloride; glass wool.

(*a*) Arrange the apparatus as in diagram (Figure 20). The U-tube should contain granulated calcium chloride.

Mix about 6 grams of *thoroughly* dried manganese dioxide with 8 grams of potassium chlorate. (C. P. quality gives a more accurate result.)

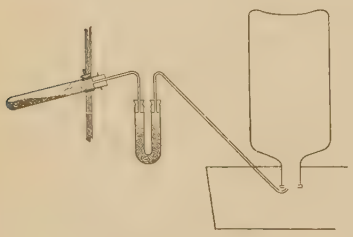


Figure 20.

Put the mixture into a dry test tube, and above the powder place a loose plug of glass wool.

Tabulate the data as indicated by the table on page 55.

(*b*) Weigh to a centigram the test tube containing the mixture, the connecting tube with its two stoppers, and the U-tube.

Record the weight in the table.

Clamp the apparatus in place, and adjust the rubber stopper carrying the delivery tube leading to the pneumatic trough. Collect the gas in an acid bottle of about 2 liters' capacity.

The test tube should be inclined at a slight angle so as to permit the spreading of the black mixture along the tube. Heat with a *small* flame, beginning at the top, and gradually working downward.

Carefully regulate the heat so that you can always count the bubbles of the oxygen. Continue the heating until the bottle is nearly full, or as long as time will allow.

When the oxygen has ceased to pass over, at once remove from the U-tube the delivery tube with its attached stopper. Allow the apparatus to cool.

(*c*) While waiting for this, measure the volume of the oxygen. Lower the large bottle in the trough so as to adjust the water levels to the same height, close the mouth of the bottle with a stopper or a glass plate, and remove the bottle from the trough, inverting it as you do so.

Find the volume of the oxygen by the amount of water necessary to fill the bottle, pouring the water into the bottle from a graduate.

Record in the table.

(*d*) When the test tube has cooled until it feels barely warm to the hand, weigh, as before, the test tube, connecting tube, U-tube, and contents.

Record the weight.

The loss of weight is the weight of the oxygen evolved.

Record the barometric pressure and the temperature of the water in the pneumatic trough.

This is approximately the temperature of the gas.

TABLE

Weight of generating and drying tube before heating	g.
Weight of generating and drying tube after heating	g.
Weight of oxygen evolved	g.
Volume of oxygen evolved under conditions of experiment	cc.
Temperature of oxygen	° C.
Barometric pressure	mm.
Aqueous tension (see table, p. 240)	mm.
Pressure of oxygen	mm.
Volume of oxygen at standard conditions	cc.
Weight of one liter of oxygen at standard conditions	g.

Calculate the volume of the oxygen at standard conditions.

Using the weight of oxygen as found, calculate, by means of a proportion, the weight of a liter (1000 *cc.*) *of oxygen at standard conditions.*

CALCULATIONS

EXPERIMENT 22

Bases

APPARATUS. Evaporating dish; glass plate, 4 × 4; stirring rod; four test tubes; glass funnel; ring-stand with one ring; bunsen burner; wire gauze with asbestos center.

MATERIAL. Metallic sodium; filter paper; red litmus paper; calcium oxide; sodium hydroxide solution, about 32 g. to 100 cc. of water; ammonium hydroxide solution, 1 to 5; hydrochloric acid, 1 to 1; ferric chloride solution, 8 g. per 100 cc.

Bases are the chemical opposites of acids. They consist of a metal united to a hydroxyl (OH) group.

(*a*) **Very active metals form bases by direct action with water.**

Take a freshly cut piece of sodium the size of a pea and completely remove the adhering oil with filter paper. Hold a square of glass vertically (to protect the face) in front of an evaporating dish containing about 10 cc. of water. Place the sodium on the water.

Describe the action.

What gas is liberated?

Determine the action of the solution on litmus and rub some of it between the fingers.

Results?

Evaporate part of the solution to dryness. The substance left in the dish is sodium hydroxide.

Describe its appearance.

Complete the equation:

$$Na + HOH \longrightarrow NaOH + _____$$

Is sodium hydroxide a soluble base?

(*b*) **Some bases can be prepared by action of the oxide of the metal with water.**

Place a gram of quicklime (calcium oxide, CaO) in a test tube and add 1 cc. of water. Warm the mixture until the action starts; remove the tube from the flame and see if there is continued action between the quicklime and the water. If not, warm the tube until an action begins and continues without the further addition of heat.

Describe the action.

How does the substance formed compare with the original lime?

This new substance is slaked lime (calcium hydroxide, $Ca(OH)_2$).

Write an equation for its formation.

Add water to the slaked lime, shake thoroughly, and allow the solid to settle. Pour off the clear liquid into another test tube, retaining the solid for part (*d*). Determine the action of this solution on litmus.

Result?

Is calcium hydroxide a soluble base? State reasons for your answer.

What common property have the solutions of sodium hydroxide and calcium hydroxide?

This is one of the characteristic properties of the solutions of metallic hydroxides (bases).

(*c*) **Other bases can be prepared by a process of precipitation.**

To 5 cc. of a hot solution of ferric chloride add 4 cc. of ammonium hydroxide.

Result ?

Complete the equation:

$$FeCl_3 + \quad NH_4OH \longrightarrow Fe(OH)_3 + \underline{\qquad}$$

Filter the solution and thus obtain the ferric hydroxide on a filter paper. Wash it thoroughly, using three separate portions of water. Allow each portion of the water to drain through *completely* before the next is added.

Transfer the washed precipitate to a test tube, add 5 cc. of water, and shake the mixture thoroughly. Allow the precipitate to settle and pour off most of the clear liquid. This is to complete the washing of the ferric hydroxide. Add 5 cc. of water, shake the mixture, and determine its action on litmus.

Is there any evidence that ferric hydroxide is soluble ?

Retain the contents of the tube for part (*d*).

What characteristic must bases have in order to act on litmus ?

(*d*) **Determination of the action of bases with acids.**

Place in one test tube sodium hydroxide solution, in a second, some of the solid calcium hydroxide from part (*b*), and in a third, the ferric hydroxide from part (*c*). To each add 5 cc. of hydrochloric acid. Determine whether heat is produced in each case.

Results ?

Do you observe any other evidences of chemical action ?

Complete the equations :

$$NaOH + HCl \longrightarrow _____ + _____$$

$$Ca(OH)_2 + HCl \longrightarrow _____ + _____$$

$$Fe(OH)_3 + HCl \longrightarrow _____ + _____$$

TABLE

Name of Base	Soluble or Insoluble	Action on Litmus

EXPERIMENT 23

Alkalies

APPARATUS. Six test tubes; two beakers, 150 cc.; evaporating dish; ring-stand with ring; wire gauze with asbestos center; bunsen burner; glass rod.

MATERIAL. Washing soda; borax; sodium sulphate; baking soda; solution of sodium hydroxide, 10 g. to 100 cc.; solution of ammonium hydroxide, 1 to 3; pieces of cloth with small grease spots of butter; cotton cloth; woolen cloth; red litmus paper.

The term *alkali* is applied to any substance whose water solution turns litmus blue. The soluble bases are strong alkalies, but solutions of many other substances also produce the same change in the color of litmus.

(*a*). **Alkaline reactions.**

Dissolve a little of each of the following substances in water, and test the action of its solution on litmus: washing soda, Na_2CO_3; borax, $Na_2B_4O_7$; sodium sulphate, Na_2SO_4; baking soda, $NaHCO_3$.

Results in each case?

Are any of these substances bases?

Give reasons for your statement.

Name those that are alkalies.

Why are they alkalies?

Alkalies which are not bases produce their effect on litmus because, on being dissolved, they react in a slight degree with

water, and form a very small quantity of base. The acid that is produced at the same time is relatively much weaker than the base.

Complete the equations:

$Na_2CO_3 + \quad H_2O \longrightarrow \quad NaOH + _____$

$NaHCO_3 + \quad H_2O \longrightarrow \quad NaOH + _____$

(*b*) **Alkalies as solvents for grease.**

Put into a beaker a piece of cotton cloth, on which a very small grease spot has been made with a little butter. Add sodium hydroxide solution, and boil the contents for several minutes. Remove the cloth and examine it to see if the grease spot has been affected.

Result?

Using a solution of borax as the alkali, repeat the experiment.

Result?

Which of the two alkalies has the greater grease-dissolving power?

(*c*) **Actions of alkalies on cotton and on woolen goods.**

Put a small piece of each kind of goods into separate test tubes, add sodium hydroxide solution, and boil the contents for several minutes.

Results?

Which kind of goods is the more affected by the strong alkali?

(*d*) **Testing alkalies for volatility.**

A volatile substance is one which turns completely into a gas at ordinary temperatures, or with slight heating. Put 5 to 10 drops of sodium hydroxide solution into an evaporating dish, and heat until no liquid remains.

Is there a residue ?
Is sodium hydroxide volatile ?

In a similar way, heat a few drops of a solution of washing soda.

Is this alkali volatile ?

By a third test, determine whether a solution of ammonium hydroxide is volatile ?

Result ?

If these three alkalies were applied to clothing, which would evaporate and which would remain on the cloth ?

Which of them would be most desirable to use in cleaning a grease spot or removing an acid stain from clothing ?

Give reasons for your answer.

EXPERIMENT 24

Neutralization

APPARATUS. Two test tubes; glass stirring rod; porcelain evaporating dish, 3″; notched cork to fit acid bottles; wire gauze with asbestos center; ring-stand with one ring; bunsen burner; glass plate.

MATERIAL. Sodium hydroxide; blue litmus paper; red litmus paper; hydrochloric acid, 1 to 10; potassium hydroxide; nitric acid, 1 to 10.

(*a*) Put a small piece of sodium hydroxide (about half a gram) into a test tube half filled with water. As soon as the sodium hydroxide has dissolved, fill the tube with water, and thoroughly mix the solution by pouring it back and forth from one test tube to another several times. Now pour half of the solution down a glass stirring rod into a porcelain evaporating dish (Figure 21).

Figure 21.

Lay a piece of blue litmus paper and a piece of red litmus paper on a glass plate. Touch each kind of paper with the end of the stirring rod wet with the solution.

What change do you observe?

Solutions producing such a change are called _____

Take a bottle of dilute hydrochloric acid and close the mouth of the bottle with a notched cork stopper as shown in Figure 22. Wet the end of the glass stirring rod with the dilute acid, touch the wet end to the inner surface of the dish, so that most of the liquid runs off and then touch each kind of litmus paper with the wet end of the rod.

Result?

This reaction is characteristic of all water solutions of _____

Add the acid, a few drops at a time, to the solution of sodium hydroxide in the evaporating dish. Stir the liquid thoroughly after each addition of acid, and, after stirring, touch a piece of blue litmus paper with the wet end of the stirring rod. Continue the addition of acid until you observe a change in the color of the litmus paper.

Figure 22.

The liquid now gives an _____ *reaction.*

Pour a little of the sodium hydroxide solution remaining in the test tube into the evaporating dish, stir the liquid thoroughly, and touch a piece of each kind of litmus paper with the wet end of the stirring rod. If no change in the color of the red litmus paper takes place, add a little more of the sodium hydroxide solution.

The liquid now gives an _____ *reaction.*

Now add the acid, a drop at a time, until the resulting liquid changes the color of neither blue nor red litmus paper. The solution is now neutral.

Evaporate the liquid by setting the evaporating dish on a wire gauze over the flame of a bunsen burner.

What is the color of the residue?

Taste the residue.

What is it?

Complete the equation:

$$NaOH + HCl \longrightarrow H_2O + _____$$

(*b*) Dissolve about half a gram of potassium hydroxide in a test tube full of water, and, proceeding as in (*a*), neutralize the solution with dilute nitric acid.

Evaporate the neutral solution to dryness. The residue is potassium nitrate, KNO_3.

Write the equation for the neutralization.

Potassium nitrate is called a salt, that is, it is a compound formed by the combination of a metal with an acid radical (an acid minus its replaceable hydrogen).

What radical (group of elements that tend to cling together during a chemical change) is present in every base?

What element is contained in every acid?

What becomes of the characteristic radical of a base and the characteristic element of an acid during neutralization?

What becomes of the remainder of the base and the remainder of the acid?

Complete the general statement concerning neutralization:

$$\text{acid} + \text{base} \longrightarrow \underline{\qquad\qquad} + \underline{\qquad\qquad}$$

Complete the following equation used to represent the dissociation of sodium hydroxide dissolved in water:

$$NaOH \rightleftharpoons Na^+ + \underline{\qquad\qquad}$$

Which of these ions is present in a water solution of any base?

Write the equation representing the dissociation of hydrochloric acid.

What ion is present in a water solution of any acid?

What compound results from the combination of the ion characteristic of acids with the ion characteristic of bases?

Write the reversible equation for the reaction between the metallic ion of the base and the negative ion of the acid.

EXPERIMENT 25

Determination of the Concentration of a Solution by Titration

APPARATUS. Two burettes; beaker or Erlenmeyer flask; stirring rod; ring-stand with two clamps.

MATERIAL. Solutions of hydrochloric acid (preferably fifth-normal, made by dissolving 17 cc. of concentrated hydrochloric acid in 500 cc. of water, and then making up the volume to 1000 cc.), sodium hydroxide, and phenolphthalein (made by dissolving 1 gram of phenolphthalein in 100 cc. of 50 % alcohol). A normal solution of an acid contains 1 gram of replaceable hydrogen per liter. A normal solution of a base contains 17 grams of replaceable hydroxyl per liter.

Fill one burette above the zero mark with a solution of hydrochloric acid of known concentration. Draw off enough of the acid to remove the air bubbles from the tip and bring the meniscus (curved surface of the water) to the graduated portion of the burette.

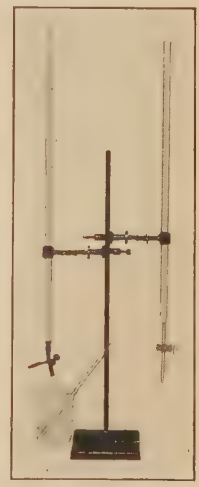


Figure 23.

Burettes are *marked* in various ways. Notice on those you have, whether each cubic centimeter is numbered, and whether the fractions are fifths or tenths of a cubic centimeter.

In reading a burette, read from the bottom of the meniscus, using care to have the eye, graduation, and lowest part of the meniscus on the same level.

Similarly, fill another burette with the sodium hydroxide solution whose concentration is to be determined and adjust the level of the liquid.

Record the readings of both burettes in a table like that given below.

Allow about 10 cc. of the sodium hydroxide solution to flow from the burette into an Erlenmeyer flask or into a beaker, and

add a *drop or two* of some indicator, *e.g.* a solution of phenolphthalein.

What color is produced when phenolphthalein is added to an alkaline solution?

To an acid solution?

Why is phenolphthalein called an indicator?

Allow the hydrochloric acid to flow, a few drops at a time, into the sodium hydroxide solution, stirring or shaking after each addition, until the reddish tinge just disappears. Now add the sodium hydroxide solution, a drop at a time, until a reddish tinge is produced; then determine whether a drop of acid will make the solution change. If it will not, continue in the manner indicated until a change of color is produced by a drop or two of either acid or of base.

Read the burettes to tenths of a cubic centimeter and record the final readings in the table.

Make three separate determinations, washing out the flask after each determination.

TABLE

	Determination.			
	1	2	3	Aver.
Reading, acid burette (before neutralization)	...	...	...	
Reading, acid burette (when neutralization is complete)	...	...	...	
Volume of hydrochloric acid used	...	...	...	...
Reading, base burette (before neutralization)	...	...	...	
Reading, base burette (when neutralization is complete)	...	...	...	
Volume of sodium hydroxide used	...	...	...	...

Make all calculations on the lower half of the page.

From the number of grams of hydrogen chloride in the standard hydrochloric acid used, calculate the weight of hydrogen chloride in the average volume of the acid used. Ans. _____ *g.*

From the equation

$$NaOH + HCl \longrightarrow _____ + _____$$

calculate the weight of the sodium hydroxide needed to neutralize the hydrogen chloride contained in the average volume of hydrochloric acid used. Ans. _____ *g.*

From the average volume of sodium hydroxide used, calculate the weight of sodium hydroxide in 1 *cc. of the solution tested. Ans.* _____ *g.*

Calculate the weight of sodium hydroxide in one liter of this solution. Ans. _____ *g.*

Proceeding as above, you could now make use of the sodium hydroxide solution, whose concentration is now known, to determine the concentration of a solution of sulphuric or other acid. The table and calculations would be similar to those already used.

CALCULATIONS

EXPERIMENT 26

Types of Chemical Change.

Direct Combination. Simple Decomposition.

APPARATUS. Asbestos mat; hard glass test tube; bunsen burner; electrolysis apparatus (one for entire class) like that shown in Figure 24, consisting of a U-tube provided with two side-arm delivery tubes, and with carbon electrodes; crystallizing dish, 5 in.; two 4 in. test tubes; forceps.

MATERIAL. Iodine; yellow phosphorus; filter paper; mercuric oxide; hydrochloric acid, concentrated; wooden splinter; saturated solution of common salt.

Relation of energy to chemical action.

Energy is the ability to do work. The more common ways in which it manifests itself are through heat, light, and electricity.

Name chemical reactions in which these forms of energy are concerned.

(*a*) Direct combination.

Put a small, thin piece of yellow phosphorus on a piece of filter paper on an asbestos mat. **Phosphorus must be kept under water until the moment of use. It takes fire easily, and its burns are serious.** With forceps, lay a crystal of iodine on the piece of phosphorus. *Stand well back*, as the action is vigorous.

Are phosphorus and iodine elements or compounds?

Complete the equation:

$$____ + ____ \longrightarrow PI_3.$$

Why is the reaction called a direct combination?

Is energy liberated or absorbed in this reaction?
State reasons for your answer.

Name three other experiments that you have performed in which elements combined directly without continuous application of heat.

State in each case whether, after the action was once started, energy was absorbed or liberated.

This evidence will guide you in choosing a word to insert in the blank in the statement of the following important principle:

Direct combinations take place readily only when energy is _____

(*b*) **Direct decompositions.**

Heat a little mercuric oxide in a hard glass test tube. Test for oxygen from time to time.

What forms on the sides of the tube ?

Write an equation for the reaction.

Why is the action called a direct decomposition ?

Stop heating the mercuric oxide.

Does the action continue ?

Is energy liberated or absorbed in this action ?

The instructor should have in operation one or more of the pieces of apparatus like that in Figure 24. In this an electric current is passing through a concentrated solution of hydrochloric acid. The gas that is given off from the anode is collected over a saturated solution of common salt.

What gas is given off at the anode ?
At the cathode ?

Stop the flow of the current.

Does the action continue ?

Is energy liberated or absorbed in this reaction ?

Write an equation for the reaction.

Why is it called a direct decomposition ?

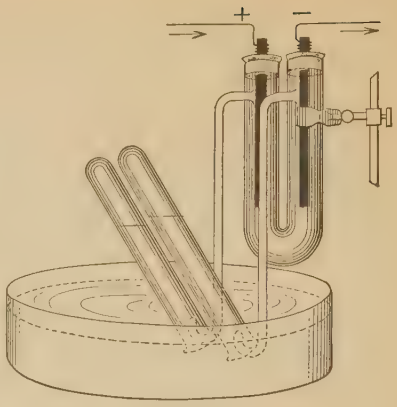


Figure 24.

The evidence of these two experiments will guide you in choosing a word to fill in the blank in the statement of the following principle :

*In most cases of direct decomposition, energy is*_____.

EXPERIMENT 27

Types of Chemical Change. Simple Replacement

APPARATUS. Two test tubes.

MATERIAL. Zinc; dilute hydrochloric acid, 1 to 4; fine iron filings; saturated solution of copper sulphate.

(*a*) Place a piece of zinc in a test tube and add 5 cc. of dilute hydrochloric acid. After the action has continued for several minutes, feel the test tube.

Is energy liberated or absorbed in the reaction?

Write an equation for the reaction.

Why is this said to be a case of simple replacement?

(*b*) To 8 cc. of a saturated solution of copper sulphate contained in a test tube, add 2 cc. of fine iron filings. Close the mouth of the tube with the thumb and shake the solution back and forth several times. After the action has continued a few minutes, feel the test tube.

Is energy liberated or absorbed in the reaction?

Allow the test tube to stand for several minutes.

What change takes place in the color of the solution?

What change takes place in the appearance of the solid?

Complete the equation:

$$CuSO_4 + Fe \longrightarrow _____ + _____$$

Why is this called a case of simple replacement?

From the evidence of the two experiments, choose a word to fill in the blank in the following statement of an important principle:

When simple replacements take place, energy is _____.

Heats of formation.

In the case of most compounds, energy is liberated when the elements unite to form the compound. The amount of energy is known as the *heat of formation.* In the case of certain compounds energy must be furnished, as it is absorbed when the compound is formed. In such a case, the substance is said to have a negative heat of formation. The amount of energy is expressed in calories of heat. A calorie is the amount of heat necessary to warm one gram of water one degree centigrade. The heat of formation is the number of calories of heat absorbed or liberated during the formation of one gram-molecule (a weight in grams equal to the molecular weight) of a compound from its elements.

The following table gives the heats of formation of certain compounds.

TABLE

HEATS OF FORMATION OF CERTAIN COMPOUNDS

	CALORIES		CALORIES
Calcium aluminum silicate	1195550	Magnesium (chloride) (dil. sol.)	187100
Calcium carbide	−6250	Magnesium sulphate (dil. sol.)	321100
Carbon disulphide	−19000	Mercuric chloride (dil. sol.)	50300
Copper (cupric) chloride (dil. sol.)	62500	Mercuric cyanide	−62500
Copper (cupric) sulphate (dil. sol.)	197500	Nitrous oxide	−20600
Hydrogen chloride	22000	Nitric oxide	−21600
Hydrogen bromide	8400	Phosphorus pentoxide	369400
Hydrogen iodide	−7000	Potassium iodide (dil. sol.)	81800
Hydrogen sulphide	4800	Potassium bromide (dil. sol.)	90400
Iron carbide	8460	Potassium chlorate	93800
Iron (ferrous) chloride (dil. sol.)	100100	Potassium chloride (dil. sol.)	101200
Iron (ferric) chloride (dil. sol.)	255700	Silicon carbide	1963
Iron (ferrous) sulphate (dil. sol.)	234900	Silver oxide	7000
Iron (ferric) sulphate (dil. sol.)	650500	Sodium chloride (dil. sol.)	96900
		Sodium iodide (dil. sol.)	70400
		Zinc chloride (dil. sol.)	113300
		Zinc cyanide	−27900

Bearing in mind the first principle stated in Experiment 26, pick out from the table five compounds that could easily be formed by direct combination.

Name three that could not be formed easily by direct combination.

In decomposing a compound, exactly as much energy must be furnished as is liberated when the compound is formed.

Bearing this fact in mind, and also the second principle stated in Experiment 26, name five compounds that it would be difficult to decompose.

Also name five that would easily be decomposed.

Name three that would decompose with liberation of energy.

To decide whether or not replacement actions will take place, compare the heats of formation of the original compound with that of the one that might be formed in the reaction.

What principle will, then, guide you in making your decision?

Of the following equations, complete those which you think will actually occur; in the other cases write the words "no reaction."

$ZnCl_2 + Cu \longrightarrow$ _____ + _____.

$KBr + Cl_2 \longrightarrow$ _____ + _____.

$NaCl + I \longrightarrow$ _____ + _____.

$CuCl_2 + Mg \longrightarrow$ _____ + _____.

$HgCl_2 + Cu \longrightarrow$ _____ + _____.

Verify your conclusions in one or two cases.

EXPERIMENT 28

Types of Chemical Action. Double Decompositions.

APPARATUS. Six test tubes.

MATERIAL. Solutions of barium nitrate, lead nitrate, silver nitrate, ammonium chloride, sodium sulphate, sodium chloride, copper sulphate (all approximately N/5), sodium hydroxide (1 to 10), dilute hydrochloric acid (1 to 3); solid sodium sulphite, copper sulphate, sodium carbonate, ferrous sulphide, ammonium chloride.

(*a*) **Effect of insolubility of one of the products.**

To 5 cc. of a solution of barium nitrate, add a little of a solution of sodium sulphate.

What evidence is there that chemical action has occurred?

Allow the tube to stand a few minutes. The substance that settles is barium sulphate.

Show by completing the following equations what ions are formed by barium nitrate and by sodium sulphate:

$$Ba(NO_3)_2 \rightleftharpoons _____ + _____$$

$$Na_2SO_4 \rightleftharpoons _____ + _____$$

Explain why these actions are reversible reactions.

Show by an equation how barium sulphate results on mixing the two solutions.

In this case, is there equilibrium, or an action that goes to an end?

Explain why such an action is called a double decomposition.

The remaining parts of the experiment are intended to illustrate the conditions under which, on mixing two substances that ionize, we get either an equilibrium or an action that goes to an end.

Try the action between solutions of the substances paired in the following table; record your observations of the results in column 2; the information needed for column 5 may be found in the table on page 236; fill in the last column after studying the rest of the table as a whole:

TABLE

Substances Whose Solutions are Mixed	Does Action go to an End or Remain in Equilibrium	Ions Existing in Original Solutions	Products Possible by New Combinations of Ions	Possible Products Soluble or Insoluble? (See p. 236)	Reason for Action (in Case it Goes to an End)
$AgNO_3 + NaCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$Pb(NO_3)_2 + Na_2SO_4$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$CuSO_4 + NaOH$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$NH_4Cl + Na_2SO_4$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$Pb(NO_3)_2 + NaCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$Ba(NO_3)_2 + NaCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$CuSO_4 + NH_4Cl$			(*a*)	(*a*)	
			(*b*)	(*b*)	

Complete the following sentence: A double decomposition will go to an end if one of the possible products is _____.

(*b*) **Effect of volatility of one of the products.**

In these experiments, use the first-named substance as a solid. Fill the curved bottom of the tube with the substance and add

about 2 or 3 cc. of the second compound. Fill in the table as in part (*a*). Information for column 5 may be found on page 237.

TABLE

Substances Used	Does the Action Go to an End?	Ions that Could be Formed	Products Possible by New Combination of Ions	Possible Products Volatile? (See p. 237)	Reason in Case Action Goes to an End
$Na_2SO_3 + HCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$CuSO_4 + HCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$Na_2CO_3 + HCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$FeS + HCl$			(*a*)	(*a*)	
			(*b*)	(*b*)	
$NH_4Cl + NaOH$			(*a*)	(*a*)	
			(*b*)	(*b*)	

Complete the following sentence: Double decompositions go to an end if one of the possible products is _____.

It will be seen from these experiments that a double decomposition goes to an end if one of the products *leaves the field of action.* If water is found in a double decomposition, it also leaves the field of action, because water does not form ions readily, and therefore is as much out of the action as if it were insoluble or volatile.

Explain why a reaction between a base and an acid goes to an end.

EXPERIMENT 29

Salts that are not Neutral.

APPARATUS. Two test tubes.

MATERIAL. Sodium carbonate; copper sulphate; ferric chloride; dilute solutions of aluminum sulphate, potassium chloride, borax, potassium nitrate, zinc sulphate, and ammonium sulphide; red and blue litmus papers.

(*a*) Fill the curved portion of a test tube with powdered sodium carbonate, Na_2CO_3. Add water till the tube is two thirds full and shake it until the sodium carbonate is dissolved. Into the solution dip a strip of blue litmus and a strip of red litmus paper.

Record the result in the tabular form given on page 81.

Water is *very slightly* dissociated into its ions according to the equation:

$$HOH \rightleftharpoons H^+ + OH^-$$

Write the equation for the dissociation of sodium carbonate.

What four kinds of ions are present in the solution?

Complete the following equilibria:

$$2\,H^+ + CO_3^{--} \rightleftharpoons _____$$

$$Na^+ + OH^- \rightleftharpoons _____$$

Carbonic acid has only a slight tendency to ionize, while sodium hydroxide ionizes in much greater degree. Therefore the first of these two actions tends to remove from the solution the H^+ ions of the water in greater degree than the second tends to remove the OH^- ions.

Which of these two ions tends to remain in excess?

How does this explain the action of sodium carbonate solution on litmus?

The practical effect of dissolving sodium carbonate in water is to form, in slight degree, undissociated carbonic acid and dissociated sodium hydroxide.

Write an equation showing this.

Of what process is the equation the reverse?

Complete the statement:

Sodium carbonate is formed by neutralizing a _____ (strong or weak) acid with a _____ (strong or weak) base. Such a salt gives an _____ reaction in water solution.

Similarly test a solution of sodium tetraborate (borax) with the litmus papers.

Record the results in your table.

Why would you expect sodium tetraborate to give such a reaction?

(*b*) In a similar manner prepare solutions of copper sulphate and ferric chloride, $FeCl_3$. Test each with red and blue litmus.

Record the results in your table.

In each case, name the base formed that has little tendency to dissociate again.

Write the equation for the formation of each of these two bases from their ions.

What ion is responsible for the litmus reaction obtained?

Write the equations showing the combination of this ion with the non-metallic ion of the two salts.

Write the equation showing the practical effect of dissolving copper sulphate in water.

Write a similar equation for the dissolving of the ferric chloride.

Of what process is the action in each case the reverse?

Complete the statement:

Salts with an acid reaction are formed by neutralizing a _____ (strong or weak) acid with a _____ (strong or weak) base.

(*c*) Aluminum hydroxide and zinc hydroxide are weak bases. Hydrosulphuric acid, H_2S, and tetraboric acid are weak acids.

Make a prediction as to the effect on litmus to be expected from solutions of each of the following salts, — aluminum sulphate, potassium chloride, potassium nitrate, zinc sulphate, and ammonium sulphide. Test your predictions by the use of litmus with a solution of each.

Record the results in your tabular form.

Complete:

weak acid + strong base ⟶ salt with _____ reaction,
strong acid + weak base ⟶ salt with _____ reaction,
strong acid + strong base ⟶ salt with _____ reaction.

Salt	Action with Blue Litmus	Action with Red Litmus
Sodium carbonate		
Copper sulphate		
Ferric chloride		
Aluminum sulphate		
Potassium chloride		
Sodium tetraborate		
Potassium nitrate		
Zinc sulphate		
Ammonium sulphide		

EXPERIMENT 30

Flame Tests

APPARATUS. Bunsen burner; three cobalt glass plates. Each solution should be contained in a small bottle or vial with a cork stopper, through which passes a glass tube carrying a platinum, iron, or nichrome wire.

MATERIAL. Solutions of salts of lithium, sodium, potassium, calcium, strontium, and barium; mixed solutions of sodium and potassium salts; unknowns.

Figure 25.

(*a*) Test salts of lithium, sodium, potassium, calcium, strontium, and barium. Hold one platinum wire at a time in the hot outer portion of a bunsen flame (Figure 25). Observe the color of the flame in each case. *Record result in tabular form.* Be careful to replace each wire in its special bottle.

TABLE (PART *a*)

SOLUTION	FORMULA	COLOR OF FLAME

The characteristic coloration of the flame in each case is due to the vapor of the metal contained in the compound.

(*b*) Observe the color of a sodium flame through three thicknesses of cobalt glass. In a similar way examine the potassium flame through the cobalt glass.

Record the result of each flame test in tabular form as indicated below.

Can you see the sodium flame through the cobalt glass?
What is the effect of the cobalt glass on the potassium flame?

Take a solution of a mixture of sodium and potassium salts, and, without using the cobalt glass, note the flame color.

Why are not both the characteristic colors seen?

Use the cobalt glass with the mixed solution and find which flame can be recognized.

What is the use of the cobalt glass in making flame tests of mixtures of sodium and potassium salts?

What use might be made of the flame tests in analytical work?

TABLE (PART *b*)

SOLUTION	COLOR — NAKED EYE	COLOR — COBALT GLASS

(*c*) Test an unknown salt obtained from the instructor. When in doubt regarding the unknown, verify your conclusion by placing the wire dipped in the unknown in one side of the flame, and, in the other side, a wire dipped in a solution of a salt of the metal you think is present.

Record the resulting color, and state the metal present.

Have your conclusion checked by the instructor.

EXPERIMENT 31

Preparation of an Acid Salt

APPARATUS. Graduate, 50 cc.; two beakers; stirring rod; ring-stand with large ring; wire gauze with asbestos center; bunsen burner.

MATERIAL. Solutions of sulphuric acid (1 to 5), and potassium hydroxide (200 grams to the liter); litmus paper.

(*a*) Take 25 cc. of dilute sulphuric acid and neutralize it with potassium hydroxide solution.

Write the equation.

Evaporate one third of the water in the neutral solution and set the remainder aside to crystallize.

(*b*) In another beaker again neutralize 25 cc. of dilute sulphuric acid with potassium hydroxide. Add to this neutral solution another 25 cc. of sulphuric acid. Evaporate the solution to one third of the volume and set it aside to crystallize.

Compare the crystals obtained with those obtained in part (*a*) as to size and general form.

Do they appear to be crystals of the same substance?

There are two potassium sulphates: normal potassium sulphate, K_2SO_4, and acid potassium sulphate, $KHSO_4$.

Which one was formed in part (a)?

Write the equation for the formation of the other in part (b).

Why is this called an acid salt?

EXPERIMENT 32

Preparation of Sodium Carbonate

APPARATUS. Two test tubes, one provided with a one-hole stopper and a delivery tube; ring-stand; clamp; bunsen burner.

MATERIAL. Limewater; sodium bicarbonate.

Place about two grams of sodium bicarbonate in a test tube provided with a stopper and a delivery tube that leads into a test tube containing limewater, $Ca(OH)_2$.

Heat the bicarbonate without using sufficient heat to color the flame yellow.

What collects on the inner wall of the test tube?

What produced the change in the limewater?

The solid left in the test tube is sodium carbonate.

How does the taste of the sodium carbonate compare with that of the sodium bicarbonate?

Complete the equations:

$$NaHCO_3 \text{ (heated)} \longrightarrow Na_2CO_3 + _____ + _____$$

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 + _____$$

EXPERIMENT 33

Determination of Water of Crystallization

APPARATUS. Ring-stand with two rings; pipe-stem triangle; porcelain crucible; horn pan balance; weights; bunsen burner.

MATERIAL. Crystallized barium chloride.

Barium chloride is the salt selected for this determination, because it is easily obtained pure and is neither efflorescent nor deliquescent.

Weigh a porcelain crucible on the balance. Then put into the crucible about two grams of crystallized barium chloride, and weigh carefully the crucible and its contents.

Record all weights in a tabular form like that given on page 87.

Support the crucible on a pipe-stem triangle so adjusted in height that the bottom of the crucible is a short distance above the top of the inner cone of the bunsen flame. Heat the crucible very gently at first. Too rapid heating may cause the water of crystallization to be driven off explosively, carrying along with it some of the salt. Gradually heat the crucible to the full intensity of the flame.

After fifteen minutes of this strong heating, slowly cool the crucible, and weigh the crucible and its contents.

Record the weight.

Repeat the heating and weigh again. Continue this process until you get two successive weighings with the same result. This is called "heating to constant weight."

What does this constant weight show about the water of crystallization?

From your data in the table ascertain (1) the weight of the crystallized barium chloride, (2) the weight of the anhydrous barium chloride left after heating.

The "anhydrous" salt is the crystallized salt minus its water of crystallization.

You know the weight in grams of two substances, crystallized barium chloride and anhydrous barium chloride. These weights are in the same ratio as the molecular weights of these substances.

Using the values given in the table of atomic weights (page 234), calculate the molecular weight of anhydrous barium chloride, $BaCl_2$.

Make all calculations on page 88.

Employing the three quantities that you now know, and representing the molecular weight of crystallized barium chloride by x, form a proportion in which the weights that you obtained by the balance have the same ratio as the molecular weights of the substances weighed.

Solve the proportion in the space marked "Calculations," page 88.

Find by subtraction the part of the molecular weight of the crystallized salt that is water.

How many molecules of water does this weight represent?

Write the formula of crystallized barium chloride.

Calculate the percent of water of crystallization in crystallized barium chloride.

TABLE

Weight of crucible + crystallized barium chloride	g.
Weight of crucible empty	g.
Weight of crystallized barium chloride	g.
Weight of crucible + barium chloride after *first* heating . .	g.
Weight of crucible + barium chloride after *second* heating .	g.
Weight of crucible empty	g.
Weight of anhydrous barium chloride	g.
Molecular weight of crystallized barium chloride	
Molecular weight of anhydrous barium chloride	
Molecular weight of water in crystallized barium chloride .	
Formula of crystallized barium chloride	

CALCULATIONS

EXPERIMENT 34

Forms of Sulphur

APPARATUS. Two test tubes; watch glass, $2\frac{1}{2}''$; small iron clamp for use as a test tube holder; bunsen burner; magnifying glass; pan of water.

MATERIAL. Roll sulphur; carbon disulphide; filter paper, 4″ in diameter.

(*a*) **Caution! Carbon disulphide is a volatile liquid that takes fire easily. It should never be used near a flame.**

Pour 5 cc. of carbon disulphide into a test tube. Add a piece of roll sulphur the size of a pea and shake the tube.

Pour the clear liquid into a watch glass, and set it aside to evaporate in a part of the laboratory some distance from a flame. Using a magnifying glass, examine the crystals of sulphur.

In the square provided for the purpose make a drawing of a crystal having a symmetrical form.

You have recrystallized roll sulphur under conditions that yield separate crystals. Sulphur that crystallizes as you have just observed is called *rhombic sulphur*.

(*b*) Fold a piece of filter paper as you would to fit a funnel, and lay it aside for future use. Also, have a dish of water ready for use.

Figure 26.

Half fill a test tube with small pieces of roll sulphur. Carefully melt the sulphur by holding the tube in an inclined position about four inches above a small flame (Figure 26). Rotate the tube slowly while the melting proceeds.

What is the color and consistency of the first portion of liquid obtained?

This color should be retained during the melting of all of the sulphur. The color should at no time be darker than a light amber.

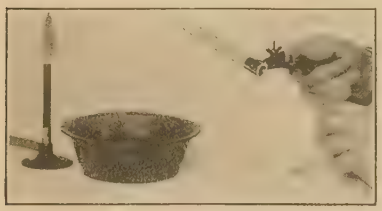


Figure 27.

Holding the folded filter paper by the edge, pour the melted sulphur into it (Figure 27). As soon as crystals have formed from the edge to the center of the surface, pour into the water in the pan that part of the sulphur that is still in a melted condition. Immediately unfold the filter paper.

Make a drawing of one of the more perfect crystals as it is seen under a microscope.

This form of sulphur is known as *prismatic sulphur*.

Keep some of the crystals for a few days and then examine them.

What changes do you observe?

Rhombic Sulphur

Prismatic Sulphur

(*c*) Half fill a test tube with small pieces of roll sulphur, and, holding the test tube with a small iron clamp, raise the temperature of the sulphur until it commences to boil. Meanwhile, tip the tube slightly from time to time and note the important changes that take place in the color and consistency (degree of fluidity) of the sulphur.

What changes in the color and consistency of the sulphur did you note from the time it melted until it commenced to boil ?

In the next operation the sulphur will probably take fire. *Do not jump.* The sulphur will burn quietly. Do not spill it on the desk. Pour the boiling sulphur slowly into cold water, keeping the mouth of the tube moving in a circle so that a thread of sulphur will form in the water (Figure 28). Examine the thread of sulphur. It is the plastic modification of sulphur.

Figure 28.

What color is it ?

Is it hard or soft ?

Elastic or brittle ?

Keep it for several days and note any change in properties.

Results?

EXPERIMENT 35

Preparation of Metallic Sulphides

APPARATUS. Test tube; ring-stand with one ring; wire gauze with asbestos center; bunsen burner; small iron clamp.

MATERIAL. Sulphur, powdered; copper, thin foil or # 30 wire; iron filings, clean and fine enough to pass through a sieve having 60 meshes to the inch; hydrochloric acid, 1 to 4; zinc dust.

(*a*) Fill the curved portion of a test tube with sulphur and heat it to boiling. Insert a strip of thin sheet copper (or fine copper wire) into the boiling sulphur.

Result ?

Withdraw the strip and compare its color, luster, and flexibility with the color, luster, and flexibility of copper.

Result ?

What is the name of the compound formed by the combination of copper with sulphur ?

Write the equation representing the formation of this compound.

(*b*) Mix thoroughly one part by volume of finely powdered sulphur with two parts of fine iron filings. Put the mixture into a test tube and heat the lower end of the tube just sufficiently to color the flame yellow. When the contents of the tube commence to glow, withdraw the flame.

Does the chemical action continue ?
Why do you think so ?

Break the tube and examine its contents.

How does it differ from sulphur in appearance ?

What gas is produced when hydrochloric acid reacts with iron?

Add *one* drop of hydrochloric acid to the substance taken from the broken test tube. Cautiously smell of the gas.

What evidence is there that the substance taken from the tube is not iron?

Write the equation for the reaction.

(*c*) This should be performed by the teacher. Mix thoroughly a pinch of sulphur with an equal bulk of powdered zinc. Placing the mixture in a conical pile on asbestos, and holding the burner *at arm's length*, cautiously ignite the pile from above.

Result?

Write an equation for this reaction.

Compare the action of copper and zinc with sulphur with the action of these metals with oxygen.

EXPERIMENT 36

Preparation and Properties of Hydrogen Sulphide

APPARATUS. Five test tubes; stopper and delivery tube to fit one of the test tubes; bunsen burner.

MATERIAL. Iron sulphide; dilute hydrochloric acid; solutions of lead nitrate, cadmium nitrate, and hydrogen peroxide; litmus paper.

(*a*) In a test tube provided with a stopper and a delivery tube place some iron sulphide and cover with dilute hydrochloric acid.

Complete the equation:

$$FeS + HCl \longrightarrow _____ + _____$$

Collect the gas by downward displacement in a dry test tube, remove the delivery tube, and light the gas in this test tube.

What two products are formed when the gas burns in this way? (Examine the tube carefully for traces of moisture before it becomes heated by the flame of the burning gas.)

How do you identify them?

What does this show concerning the composition of the gas?

(*b*) If the gas in the test tube burned *quietly*, light the gas at the end of the delivery tube. *Cautiously* smell the gas around the flame.

Are all the products formed the same as before?

State the reason for your answer.

(*c*) Place the end of the delivery tube in a test tube half filled with water and let the gas bubble through the water two or three minutes.

How does the solution taste ?

What effect has it on litmus ?

Using a small portion of the solution of hydrogen sulphide in each case, add a few drops of:

(1) a solution of lead nitrate ;

(2) a solution of cadmium nitrate.

Results?

Complete the equations :

$$H_2S + Pb(NO_3)_2 \longrightarrow _____ + _____$$

$$H_2S + Cd(NO_3)_2 \longrightarrow _____ + _____$$

The cadmium compound precipitated is used as an artist's pigment.

It is known as cadmium _____

(*d*) Allow the gas from the generator to bubble through a solution of hydrogen peroxide until a decided effect is obtained.

Results ?

Complete the equation :

$$H_2S + H_2O_2 \longrightarrow _____ + _____$$

EXPERIMENT 37

Sulphur Dioxide

APPARATUS. Flask, 250 cc. with two-hole stopper to fit; thistle tube; bent tube with short arms; bent tube with one long arm; rubber connection tube; bottle, 150 cc.; two test tubes; cover glass; glass stirring rod; ring-stand with one ring and one clamp; bunsen burner.

MATERIAL. Sodium bisulphite; sulphuric acid, 1 to 1; pink carnation; blue litmus paper; barium chloride solution, 1 to 20; hydrochloric acid, 1 to 4; dilute solution of potassium permanganate, three or four small crystals dissolved in a liter of water.

Figure 29.

(*a*) **Preparation.**

Arrange the apparatus as shown in Figure 29. Be sure that the end of the thistle tube is very near the bottom of the flask.

Pour 10 grams of sodium bisulphite ($NaHSO_3$) into the flask, then replace the stopper and pour through the thistle tube sufficient water to very little more than cover the end of the tube. Now add sulphuric acid (1 to 1), a little at a time, until chemical action starts. Other portions of acid are to be added from time to time to cause the reaction to continue. Warm the flask gently, if necessary, to increase the speed of the reaction.

Complete the equation:

$$NaHSO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + ____ + ____$$

(*b*) **Physical properties.**

Watch the size of the bubbles of gas as they rise through the water in a test tube full of cold water, into which the delivery tube has been inserted, especially after the air in the flask has been displaced.

Is sulphur dioxide soluble in water?

Set the test tube and contents aside for future use, and replace the test tube with the wide mouth bottle (Figure 29).

Is sulphur dioxide heavier or lighter than air ?
Why do you think so ?

Compare the odor of the gas with the odor you observed when you burned sulphur in oxygen, or in air.

Result ?

(*c*) **Chemical properties.**

Wet a pink carnation and put it in a bottle of sulphur dioxide. Cover the bottle and allow it to stand for a short time.

Result ?

Touch a piece of blue litmus paper with the end of a stirring rod wet with the liquid left in the test tube.

Result ?

Why is sulphur dioxide not an acid ?

An oxide that unites with water to form an acid is an *acid anhydride..*

Is sulphur dioxide such a compound ?
Complete the equation :

$$SO_2 + H_2O \longrightarrow ____$$

(*d*) **Test for SO_4^{--} ions.**

SO_4^{--} ions unite with Ba^{++} ions to form barium sulphate, a white solid insoluble in water and in dilute hydrochloric acid. This fact is commonly employed in testing for sulphuric acid or a soluble sulphate.

Add a drop or two of sulphuric acid to about 10 cc. of water in a test tube, and then add a few drops of a solution of barium chloride.

Result ?

Complete the equation:

$$H_2SO_4 + BaCl_2 \longrightarrow _____ + _____$$

Determine whether the precipitate is soluble in dilute hydrochloric acid.

Result ?

(*e*) **Oxidation of sulphurous acid.**

Add 5 cc. of the water solution of sulphurous acid to an equal volume of a dilute solution of potassium permanganate, $KMnO_4$.

Result ?

To the solution obtained add barium chloride solution and a little hydrochloric acid.

Result ?

Into what ions did the potassium permanganate convert the SO_3^{--} *ions present in the solution ?*

What term is applied to such a process ?

What element was taken from the potassium permanganate ?

Is sulphurous acid a reducing or an oxidizing agent ?

Why ?

Is potassium permanganate a reducing or an oxidizing agent ?

Why ?

EXPERIMENT 38

Preparation and Properties of Sulphur Dioxide. Reduction Method

APPARATUS. Ring-stand with one ring and one clamp; wire gauze with asbestos center; bunsen burner; 250 cc. flask with stopper carrying a doubly bent delivery tube and a thistle tube; two wide-mouth bottles, one of which is provided with a two-hole rubber stopper to fit; two bent tubes with rubber connection tube; glass plate.

MATERIAL. Copper (rivets, turnings, or small clippings); concentrated sulphuric acid; pink carnation; blue litmus paper; barium chloride solution; dilute hydrochloric acid; hydrogen peroxide solution.

(*a*) Preparation.

Arrange the apparatus as shown in Figure 30. The bottle *A* is used as a safety bottle to prevent water from being forced back into the hot concentrated sulphuric acid, in case the pressure in the flask decreases during the preparation of the sulphur dioxide. Be sure that the delivery tube from the flask does not extend more than one fourth the way down the safety bottle *A*. Take the flask and, holding it in an inclined position, allow the pieces of copper to slide down the neck of the flask, so as not to break the flask. Replace the stopper and pour through the thistle tube sufficient concentrated sulphuric acid to cover the copper. Fill one half of the second bottle with water.

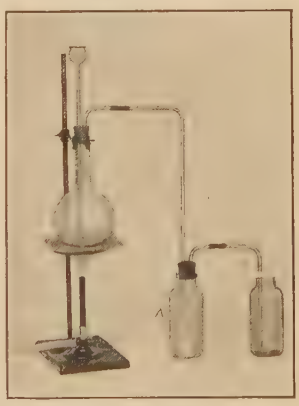


Figure 30.

Caution!! Hot concentrated sulphuric acid produces very severe burns when it comes in contact with the skin. The utmost care should therefore be exercised to avoid being burned in case the flask breaks. Keep the hand from under the flask and step back in case the flask cracks.

Heat the flask with a small flame. In no case let the flame touch the glass. Stop heating the flask as soon as a free evolution of gas is obtained.

(*b*) **Physical properties.**

If the odor of the gas is not apparent, it may be detected by wafting toward the nostrils with the hand a little of the gas as it bubbles from the bottle.

What is the name of the gas?
What gas is frequently liberated when an acid reacts with a metal?

Since hot, concentrated sulphuric acid is an oxidizing agent, what compound of this gas would be likely to be formed?

The products actually formed depend on the concentration of the sulphuric acid used, and the temperature at which the experiment is carried on.

Assuming that sulphurous acid, H_2SO_3, is the reduction product, into what compounds would it decompose at atmospheric pressure?

Complete the equation:

$$Cu + 2\,H_2SO_4 \longrightarrow CuSO_4 + _____ + _____$$

Watch the size of the bubbles of sulphur dioxide as they rise through the water in the second bottle.

Is sulphur dioxide soluble in water?
Explain.

Is sulphur dioxide heavier or lighter than air?
Why do you think so?

(*c*) **Chemical properties.**

Put a wet pink carnation into the safety bottle containing sulphur dioxide.

Result?

Test the liquid in the second bottle with blue litmus paper.

Result ?

Write an equation to explain how an acid was produced.

Why is sulphur dioxide an acid anhydride ?

What is the name of the acid of which it is the anhydride ?

Add a little hydrogen peroxide to the solution in the second bottle. Pour a little barium chloride solution into the liquid.

Result ?

Determine whether the product is soluble in dilute hydrochloric acid.

Result ?

For what ion have you just tested ?

Write the equation for the reaction between sulphurous acid and hydrogen peroxide.

Is hydrogen peroxide an oxidizing or a reducing agent ?

EXPERIMENT 39

Properties of Sulphuric Acid

APPARATUS. Beaker; two test tubes; porcelain evaporating dish; flask, 50 cc.; glass stirring rod; ring-stand with ring and small clamp; gauze with asbestos center; bunsen burner.

MATERIAL. Concentrated sulphuric acid; dilute hydrochloric acid; barium chloride solution, 1 to 20; sodium sulphate solution, 1 to 20; cane sugar; zinc strips; fine copper wire or copper gauze; wood splinter.

(*a*) Action with water.

Caution! In mixing concentrated sulphuric acid with water, the acid should be slowly poured into the water, with constant stirring. The reverse method produces a dangerously explosive spattering.

Pour a test-tubeful of water into a beaker. Into this water slowly pour one sixth of a test-tubeful of concentrated sulphuric acid, frequently stirring the mixture with a glass rod. Feel the outside of the beaker.

What noticeable effect is produced?

Keep for parts (*b*) and (*d*) the dilute sulphuric acid just prepared.

(*b*) Action with metals.

Put a zinc strip into one sixth of a test tube of concentrated sulphuric acid.

Is there much action between the zinc and the concentrated sulphuric acid?

Pour the contents of the test tube into the sink and wash down the acid with water. Rinse off the zinc strip and return it to the test tube. Then pour upon it some of the dilute sulphuric acid made in part (*a*).

Describe the action.

Name the gaseous product, and write the equation for the reaction.

Figure 31.

How does dilute sulphuric acid differ from the concentrated acid in its action with metals ?

Place some copper wire gauze or a small loosely rolled ball of fine copper wire in a small flask, and add a fifth of a test tube of concentrated sulphuric acid. Support the flask on an asbestos gauze on a ring-stand in a hood, and loosely clamp the neck of the flask (Figure 31). Heat the flask carefully *with a small flame* until action commences. Then remove the flame.

Describe the action.

Cautiously smell the gaseous product.

What is it ?

What gas is usually liberated when an acid reacts with a metal ?

Remembering that hot, concentrated sulphuric acid acts as an oxidizing agent, explain why we do not get this gas here.

From which of the original materials is the sulphur dioxide derived?

Name the salt formed.

Complete the equation:

$$Cu + 2\,H_2SO_4 \longrightarrow _____ + _____ + _____$$

Mercury and silver react similarly to copper with hot concentrated sulphuric acid.

Write the equation for the reaction in the case of silver.

(*c*) **Dehydrating action.**

Pour one sixth of a test tube of sulphuric acid into an evaporating dish. Add a little more than enough cane sugar to soak up the acid. Allow the action to continue until a decided result is obtained.

Describe the action that occurs.

Which element of the cane sugar molecules ($C_{12}H_{22}O_{11}$) *gives the color to the residue in the dish?*

What elements did the concentrated sulphuric acid remove from the cane sugar molecules?

Dip a wooden splinter into concentrated sulphuric acid.

Results?

Explain the effect of the acid upon the wood, which is mainly cellulose, a substance represented by the formula $C_6H_{10}O_5$.

Complete the statement:

When sulphuric acid acts as a dehydrating agent on compounds, it removes from them _____ and _____ as _____.

Why is sulphuric acid used in drying gases?

(*d*) **Test for a sulphate.**

To a little sodium sulphate solution in a test tube, add a few cubic centimeters of barium chloride solution.

Describe the color and character of the compound produced.

Write the equation for its formation.

Add dilute hydrochloric acid to the precipitated barium sulphate.

Result?

Using barium chloride solution and dilute hydrochloric acid, repeat the test with dilute sulphuric acid, hydrogen sulphate.

Result?

State the test for a sulphate.

DRAWING, PART (*b*)

EXPERIMENT 40

Preparation of Nitrogen

APPARATUS. Pneumatic trough; phosphorus stand (Figure 32) consisting of (*a*) cork to fit hole in shelf of trough; (*b*) # 16 copper wire; and (*c*) blackboard crayon; iron forceps; stirring rod; burner; wide-mouth bottle, 8 oz.; glass plate.

MATERIAL. Small piece of phosphorus kept under water; filter paper; wooden splinter.

Hollow a cavity in the end of a piece of blackboard crayon. Place it on the wire stand (*b*, Figure 32) and insert cork in the hole in the shelf of a pneumatic trough. Fill the trough with water to 1 cm. above the shelf. Have close at hand an 8 oz. wide-mouth bottle.

Figure 32.

a, cork; *b*, # 16 copper wire; *c*, crayon.

Caution! Yellow phosphorus should never be handled except under water.

With a pair of forceps, take a piece of phosphorus about half the size of a pea, remove the adhering water with a bit of filter paper, and place the phosphorus in the hollow of the crayon.

Touch the phosphorus with a warm stirring rod and immediately invert over it the wide-mouth bottle, and let it rest on the shelf of the trough.

Carefully note and record the results.

When the white cloud in the bottle has cleared, press the phosphorus stand from below up into the bottle. Slide the bottle to the edge of the shelf and allow the phosphorus stand to drop down into the trough.

Cover the mouth of the bottle with a glass plate, invert it, and set it on the desk.

Describe the appearance of the nitrogen.

It is a [illegible]

Introduce a lighted splinter into the bottle.

Result? It goes out

What constituent of the air was removed by the phosphorus?

oxygen

What other constituents still remain with the nitrogen?

carbon dioxide and argon

Dispose of the phosphorus stand as directed by the instructor.

Alternative Method

APPARATUS. Pneumatic trough; two wide-mouth bottles; glass plate; ring-stand with one ring and one clamp; 250 cc. Erlenmeyer flask with two-hole rubber stopper to fit; thistle tube; delivery tube; wire gauze with asbestos center; bunsen burner; test tube.

MATERIAL. Sodium nitrite, $NaNO_2$; ammonium chloride; wooden splinter.

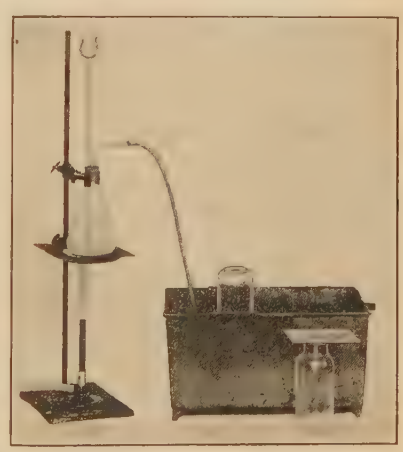


Figure 33.

Put 15 grams of sodium nitrite and 10 grams of ammonium chloride into a 250 cc. Erlenmeyer flask.

Fit the flask with a two-hole rubber stopper carrying a thistle tube and a delivery tube leading to a pneumatic trough. Support the flask on a wire gauze resting on the large ring of a ring-stand (Figure 33).

Add a test-tubeful of water to the flask through the thistle tube.

Caution!! Avoid overheating the flask, so as to prevent an explosive decomposition.

Gently heat the flask so that the nitrogen will be evolved at a temperature considerably below the boiling point of water. In case frothing occurs from overheating, pour a few cubic centimeters of cold water into the thistle tube.

After the air has been displaced from the flask, collect the nitrogen in wide-mouth bottles.

Lower a lighted splinter into a bottle of nitrogen.

Result ?

Has the nitrogen of the air an odor ?

Smell a bottle of the nitrogen collected.

What does this result show about the nitrogen collected ?

The nitrogen in the present experiment results from the decomposition of ammonium nitrite. This compound is so unstable that it is freshly prepared by the interaction of ammonium chloride and sodium nitrite.

Complete the equations:

$$NH_4Cl + NaNO_2 \longrightarrow NH_4NO_2 + ____$$

$$NH_4NO_2 \longrightarrow _____ + ____$$

DRAWING

EXPERIMENT 41

Preparation and Properties of Ammonia

APPARATUS. Ring-stand; clamp; test tube rack; three test tubes, one fitted with one-hole stopper and delivery tube; perforated cardboard square; bunsen burner; enameled pan.

MATERIAL. Slaked lime; ammonium chloride; ammonium sulphate; sodium hydroxide solution; red and blue litmus paper.

(*a*) **Preparation of ammonia.**

Take a little ammonium chloride in one hand and in the other a little slaked lime (dry). Smell of each. Rub the two together between the palms of the hands. Smell the mixture cautiously. Bring a moist strip of litmus paper near the mixture.

Results? The two have an odor ammonia turns red litmus blue

(*b*) Repeat, using ammonium sulphate and slaked lime. As before, smell, and apply litmus test.

Results? red litmus

(*c*) To a little sodium hydroxide solution in a test tube add a small amount of ammonium chloride. Heat gently, smell cautiously, and test with litmus as before.

Results? ammonia odor . turns red

Ammonium chloride, NH_4Cl, and ammonium sulphate, $(NH_4)_2SO_4$, are salts.

Regarding the reaction between sodium hydroxide and ammonium chloride as a double replacement, name the two products formed.

NaCl and NH_4OH.

Write the equation showing these two as products.

$NaOH + H_4Cl \longrightarrow NaCl + NH_4OH$

Ammonia gas, NH_3, results from the decomposition of one of the products.

Write the equation for this decomposition.

$NH_4OH \longrightarrow NH_3 + H_2O$

Complete the equation for the action in part (a).

$$Ca(OH)_2 + 2\,NH_4Cl \longrightarrow \underline{\qquad} + \underline{\qquad}$$
$$\longrightarrow \underline{\qquad} + \underline{\qquad} + \underline{\qquad}$$

Write a single equation for the action in part (b).

$Ca(OH)_2 + (NH_4)_2SO_4 \rightarrow CaSO_4 + 2NH_3 + 2H_2O$

How could you prove that a substance given you was an ammonium salt?

Figure 34.

(*d*) On a piece of paper, mix a quarter of a test-tubeful of ammonium chloride with a quarter of a test-tubeful of slaked lime. Put the mixture into a test tube provided with a delivery tube for the collection of the gas by upward displacement (see Figure 34). Warm the mixture very gently. Fill a dry test tube with the gas.

(*e*) **Properties of ammonia.**

What is the color of the ammonia gas?

white

Is this gas lighter or heavier than air?

it is lighter

(*f*) Place a test tube of ammonia mouth downward in a dish of water.

Result?

water rises in tube

What does this show?

NH_3 dissolves in water

EXPERIMENT 42

Ammonium Compounds

APPARATUS. Test tube with stopper and single-bend delivery tube; four other test tubes; ring-stand with one clamp; bunsen burner.

MATERIAL. Ammonium chloride; slaked lime; concentrated hydrochloric acid; concentrated sulphuric acid; sodium hydroxide solution, 1–10; red litmus paper; blue litmus paper; labels.

On a piece of paper, mix thoroughly 5 grams of ammonium chloride and 10 grams of dry, slaked lime. Notice the characteristic odor of ammonia. Place the mixture in a dry test tube, provided with a stopper and delivery tube. Clamp it in a nearly horizontal position with the delivery tube pointing down (Figure 35). Have at hand three test tubes: (*a*) a dry test tube to which 2 drops of concentrated hydrochloric acid have been added; (*b*) a test tube containing 1 drop of sulphuric acid; (*c*) a test tube containing water to the depth of 1 inch.

Figure 35.

(*a*) Warm gently the test tube containing the mixture, and bring the delivery tube into the test tube containing the drops of hydrochloric acid, until a solid is formed. This solid is ammonium chloride, NH_4Cl.

Write the equation for the reaction.

Was there any heat developed when it formed?

Label and reserve the material.

(*b*) Similarly use the test tube containing the single drop of sulphuric acid. The solid formed in this case is ammonium sulphate, $(NH_4)_2SO_4$.

Write the equation for the reaction.

Was there any heat developed when it formed ?

Label and reserve the material.

(*c*) In like manner, bring the delivery tube into the test tube containing water, but be particularly careful that the delivery tube does not touch the water. Observe the top of the water.

Is there any sign of action ?

Is there any heat developed ?

Test the liquid with litmus.

Result ?

Has the liquid any odor ?

The liquid is a solution of ammonium hydroxide, NH_4OH, an unstable base which is commonly used.

Write the equation for its formation.

(*d*) Using half of this solution of ammonium hydroxide, boil the liquid for three minutes, while holding a piece of moistened litmus paper across, but not touching, the mouth of the test tube.

What indication is there that something besides water vapor is escaping ?

Label and reserve the material. Ammonium hydroxide is very unstable. It decomposes, forming water and gaseous ammonia (NH_3).

Complete the equation :

$$NH_4OH \longrightarrow \quad + \quad$$

(*e*) Using the other part of the ammonium hydroxide solution obtained in part (*c*), add to it two drops of hydrochloric acid, mix well, and if there is any odor, warm the solution

gently until the odor has disappeared. Label and reserve this solution for part (*h*).

Write the equation.

$NH_4OH + HCl \longrightarrow H_2O + HCl + NH_3$

(*f*) Using the test tube containing the solid ammonium chloride (part *a*), warm it gently at the spot where the most solid seems to be.

Result ?

evaporates

Would this have happened to sodium chloride ?

no

When the test tube has cooled, add a little water.

Does the ammonium chloride dissolve ?

yes

How does it react with red and with blue litmus ?

blue

Label and reserve the material for part (*h*).

(*g*) Add a little water to the test tube containing the solid ammonium sulphate.

Does it dissolve in water ?

yes

How does it react with litmus ?

Label and reserve the material for part (*h*).

Ammonium compounds resemble chemically the compounds of potassium and of sodium. In the ammonium compounds, the group of atoms (NH_4) acts like an atom of sodium or potassium.

In this experiment, in what two cases has the ammonium hydroxide acted in the same manner as sodium hydroxide would have done ?

In d and e

In which case did it act differently ?

c

(*h*) Test for ammonium compounds.

To detect ammonium compounds, convert the compound into the hydroxide and identify the unstable ammonium hydroxide by the ammonia gas resulting from its decomposition.

$$NH_4OH \rightleftharpoons NH_3 + H_2O$$

To each of the four materials reserved, after noticing whether there is any odor, add 5 or 6 drops of sodium hydroxide solution. Notice whether there is an odor, and, while holding a piece of moistened litmus paper just above the mouth of the test tube, warm the tube gently. Observe carefully the result in each case.

Tabulate results as indicated below.

TABLE

Material in Test Tube From	Odor before Adding NaOH	Odor after Adding NaOH	Action with Litmus
d			
e			
f			
g			

Complete the equations:

$NH_4Cl + NaOH \longrightarrow$

$(NH_4)_2SO_4 + NaOH \longrightarrow$

NH_4OH heated $\longrightarrow$

$NH_4Cl + Ca(OH)_2 \longrightarrow$

Having performed the test for ammonia on these four samples, how could you now tell whether the original material was a chloride or a sulphate?

EXPERIMENT 43

Preparation and Properties of Nitric Acid

APPARATUS. Retort, 100 cc.; two test tubes; battery jar; ring-stand with large ring; wire gauze with asbestos center; clamp; bunsen burner; funnel or thistle tube; flask.

MATERIAL. Sodium nitrate; concentrated sulphuric acid; concentrated hydrochloric acid; ferrous sulphate solution, freshly prepared; copper strip; excelsior; unknowns.

Caution! Concentrated sulphuric and nitric acids are dangerous to both flesh and clothing.

(*a*) **Preparation.**

Put about 15 grams of sodium nitrate into a tubulated retort. Place the retort on a wire gauze. Insert the neck of the retort as far as possible, but not tightly, into a flask partly immersed in water (Figure 36).

Clamp the retort in position and pour 10 cc. of concentrated sulphuric acid through a funnel, or thistle tube, upon the nitrate.

Replace the stopper and heat the contents of the retort gently. Be careful not to allow the flame to pass through the gauze.

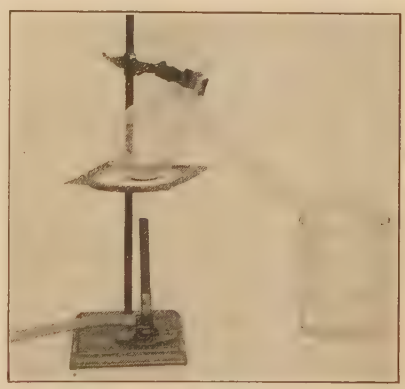


Figure 36.

What two changes of state take place in the distillation of the acid?

Distil, using moderate heat, as long as any nitric acid runs down the neck of the retort.

Allow the retort and its contents to cool without removing the retort from the stand.

Complete the equation:

$$NaNO_3 + H_2SO_4 \longrightarrow _____ + _____$$

Why does this reaction go to an end?

Upon what characteristic of sulphuric acid does its use in this experiment depend?

(*b*) **Oxidizing action of nitric acid.**

N.B. The acid collected is much more active than the ordinary nitric acid.

Use it very carefully and throw all solid materials in a waste jar immediately after examining them.

Put into a test tube 1 cc. of the acid that you have prepared, and thrust in a small, loose plug of excelsior so that it remains about an inch above the acid. Hold the test tube by means of a holder and heat the acid until it boils vigorously, and the vapor reaches the excelsior. After a moment, hold the tube so that the flame is directly under the excelsior for a few seconds.

Results?

This action is chiefly due to the very strong oxidizing character of nitric acid.

Fill the curved bottom of the test tube (1 cc.) with the acid you have prepared. Add twice the volume of hydrochloric acid and heat the mixture.

What change in color takes place?

Smell cautiously the gas issuing from the test tube.

What is this gas?

Complete the equation:

$$HNO_3 + \quad HCl \longrightarrow _____ + _____ + NO$$

Explain how this shows the oxidizing power of nitric acid.

Such a mixture of nitric and hydrochloric acids is known as *aqua regia*.

(*c*) **Action of nitric acid with metal.**

Put a small strip of copper into a test tube and add a few drops of the acid that you have prepared and a few drops of water.

Result ?

Supposing that hydrogen is at first formed, as in the typical action of an acid with a metal, what further action would occur as a result of the property of nitric acid shown in part (*b*) *?*

(*d*) **Test for a nitrate.**

Dissolve *a very small amount* of sodium (or potassium) nitrate in 2 cc. of water. Add an equal volume of ferrous sulphate solution. Hold the tube in an inclined position, and pour slowly down the side of the tube 2 cc. of concentrated sulphuric acid. The heavy acid will run down the tube and form a separate layer under the mixture of the other two solutions.

Describe the appearance between the two layers.

This is a very delicate indication of a nitrate. Repeat the test once or twice using unknown solutions furnished by the instructor.

Record the results obtained with the unknown solutions.

EXPERIMENT 44

Preparation and Properties of Nitric Oxide

APPARATUS. Four wide-mouth bottles (6 to 8 oz.); 2-hole rubber stopper to fit wide-mouth bottle; thistle tube; delivery tube; pneumatic trough or dish; oxygen generator, consisting of wide-mouth bottle (6 oz.), with a 2-hole rubber stopper carrying a delivery tube and a funnel having a glass rod with one end ground into the funnel so as to form a stopper (Figure 37); test tube.

MATERIAL. Copper (wire, rivets, or turnings); concentrated nitric acid; sodium peroxide.

(*a*) Preparation.

Arrange a wide-mouth bottle with a stopper carrying a thistle tube and delivery tube. Place in the bottle about 10 g. of copper and cover with a test-tubeful of water.

Pour about one third of a test-tubeful of concentrated nitric acid through the thistle tube of the generator and wait for the action to start. Collect the gas by the displacement of water. To maintain the action in the generator, add from time to time small quantities of the concentrated nitric acid.

Note the color of the gas that first appears in the generator.

Why does it not appear in the collecting bottle?

What is the difference in color between this gas and the one that does collect in the bottle?

The gas that collects in the bottle over water is nitric oxide, NO.

Collect one full bottle, and another bottle half full of the nitric oxide, and let them stand in the trough for use later.

Note the color of the liquid in the generator. This color is characteristic of the water solution of cupric salts.

What gas is often produced by the action of an acid with a metal?

What oxidizing action prevents our getting this gas here?

Name the product that is formed instead.

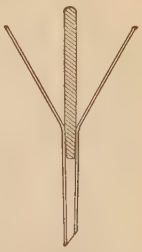


Figure 37.

The removal of oxygen from certain nitric acid molecules results in their reduction to nitric oxide and water.

Complete the equation:

$$3\ Cu + 8\ HNO_3 \longrightarrow \quad Cu(NO_3)_2 + _____ + _____$$

(*b*) **Action with oxygen.**

Obtain from the instructor one of the bottles for generating oxygen (Figure 38) by the action between water and sodium peroxide:

$$2\ Na_2O_2 + 2\ H_2O \longrightarrow 4\ NaOH + O_2$$

Figure 38.

Gently loosen the stopper in the funnel so as to allow a *few* drops of the water in the funnel to fall upon the sodium peroxide at the bottom of the generator.

Allow the air in the delivery tube to be displaced by the oxygen. If the action stops, let another drop of water fall on the peroxide.

Caution! To avoid violent action, use water in small amounts with intervals between.

Pass oxygen, a little at a time, by means of the delivery tube, into the bottle that is half full of nitric oxide, which is standing in the pneumatic trough.

What are the two noticeable results?

In what respect does this colored gaseous product differ in composition from the nitric oxide?

Complete the equation:

$$2\ NO + O_2 \longrightarrow ____$$

(*c*) **Action with air.**

Allow the full bottle of nitric oxide to escape into the air.

What evidence of a chemical change do you observe?

With what gas in the air does the nitric oxide combine?

Account for the reddish brown gas that appeared in the generator at the beginning of part (a).

Compare the solubility in water of nitric oxide and of nitrogen peroxide.

DRAWING

EXPERIMENT 45

Preparation and Properties of Nitrous Oxide

APPARATUS. Flask and small bottle, with stoppers and delivery tubes as shown (Figure 39); ring-stand; clamp; bunsen burner; three bottles (6 oz.); dish of water; glass plate; test tube, fitted with rubber stopper carrying delivery tube.

MATERIAL. Ammonium nitrate; anhydrous copper sulphate; splinter; copper turnings; concentrated nitric acid.

(*a*) Put 10 grams of pure crystallized ammonium nitrate into the flask and arrange the apparatus as shown in Figure 39. Heat the flask very cautiously, keeping the flame in constant motion. If brown fumes appear in the flask during the heating, allow the flask to cool a little.

Caution! The brown fumes indicate a decomposition that may become explosively violent.

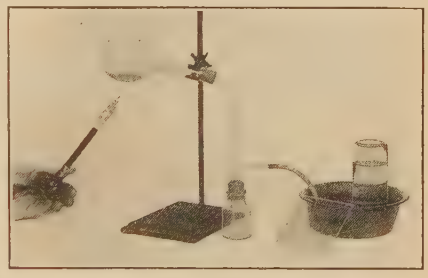


Figure 39.

Collect one bottle and two half bottles of nitrous oxide by the displacement of water. Leave the half-filled bottles standing in water until needed for parts (*b*) and (*d*).

Drop a little anhydrous copper sulphate into the small bottle.

What liquid is in the bottle?

Complete the equation:

$$NH_4NO_3 \longrightarrow N_2O + \text{____}$$

Has the gas an odor?

(*b*) Place the palm of the hand over the mouth of the half-filled bottle. Press down *tightly* and shake vigorously.

Result?

Explain.

(*c*) Lower a glowing splinter into a bottle of nitrous oxide.

Result?

(*d*) Place small pieces of copper turnings in a test tube provided with stopper and delivery tube. Cover the copper with nitric acid, immediately insert the stopper, and place the end of the delivery tube under the water of the dish. As soon as colored gas no longer shows in the test tube generator, insert the end of the delivery tube under the mouth of one of the bottles that was half filled with nitrous oxide.

Does nitrous oxide react with nitric oxide in the same way that oxygen did in Experiment 44?

How could you determine whether a gas was nitrous oxide or oxygen?

DRAWING

EXPERIMENT 46

Preparation and Properties of Bromine

APPARATUS. Ring-stand; clamp; four test tubes; one-hole stopper and a delivery tube; beaker; bunsen burner; test tube rack.

MATERIAL. Potassium bromide; manganese dioxide; sulphuric acid, 2 to 1; carbon disulphide; chlorine water.

Caution! Keep flames away from carbon disulphide. Its vapor is explosive when mixed with air.

(*a*) Preparation.

On a piece of paper, mix 1 gram of potassium bromide with an equal bulk of manganese dioxide.

Fit a test tube with a one-hole stopper carrying a delivery tube. Pour about 3 cc. of sulphuric acid (2 to 1) into the test tube, and add the mixture of potassium bromide and manganese dioxide. Clamp the tube containing the mixture so that the delivery tube shall extend to the bottom of an empty test tube, standing in a beaker of water (Figure 40). Warm the test tube containing the mixture very gently.

Figure 40.

What is the color of the bromine vapor and of the liquid bromine?

Nearly fill the test tube containing the bromine with water. Save the mixture for future use.

Is bromine heavier or lighter than water?

State reason for your answer.

What compounds would we expect to have formed by the action of potassium bromide with sulphuric acid?

Complete the following equations, and draw lines through the products that do not remain as permanent products of the reaction:

$$2\,KBr + H_2SO_4 \longrightarrow$$
$$MnO_2 + H_2SO_4 \longrightarrow MnSO_4 +$$

Show by an equation how the two temporary products that you have crossed out react with each other:

Show the final products of the reaction by completing the following equation:

$$2\,KBr + MnO_2 + 2\,H_2SO_4 \longrightarrow$$

(*b*) **Solubility of bromine.**

Add a few drops of carbon disulphide to a test tube one third full of water, and shake the contents.

Are the two liquids miscible: that is, are they completely soluble in each other?

To what extent is bromine soluble in water?

(*c*) Add a few drops of the bromine water obtained in part (*a*) to the mixture of carbon disulphide and water. Shake the resulting mixture vigorously and then allow it to stand for a short time.

What color does the bromine impart to the carbon disulphide?

Does all of the bromine dissolve in the carbon disulphide?

Is bromine more soluble in water or in carbon disulphide?

(*d*) **Test for a bromide.**

Dissolve a small crystal of potassium bromide in 2 or 3 cc. of water, add a little carbon disulphide, and shake the mixture. Save the resulting mixture for part (*e*).

Does bromine, when combined with other elements, color carbon disulphide ?

no

In what state must the bromine be to give the test with carbon disulphide ?

gas

(*e*) To the solution obtained in part (*d*) add a few drops of chlorine water and shake the mixture.

What evidence is there that bromine has been set free by action of the chlorine ?

Complete the equation :

$$KBr + Cl_2 \longrightarrow \underline{\quad KCl \quad} + \underline{\quad\quad}$$

What name is given to this type of chemical reaction ?

Which has the greater heat of formation, potassium bromide or potassium chloride (see Experiment 27) ?

Didn't have it

Give a test for a bromide.

chlorine DRAWING

EXPERIMENT 47

Preparation and Properties of Iodine

APPARATUS. 7 test tubes; test tube rack; bunsen burner; paper, 15 × 5 cm.

MATERIAL. Potassium iodide; manganese dioxide; sulphuric acid, 2 to 1; alcohol; potassium iodide solution; chloroform or carbon disulphide; chlorine water; bromine water; wooden splinters.

(*a*) Preparation.

Mix on a piece of paper 1 gram of potassium iodide with one-fourth its bulk of manganese dioxide. Roll the paper into a cylinder and insert it with its contents into a dry test tube held horizontally. Raise the tube to a vertical position, so that the mixture will fall to the bottom without touching the sides. Withdraw the paper, and add 2 cc. of sulphuric acid (2 to 1). Warm the contents of the tube very gently.

Results?

Describe the deposit of iodine.

Complete the following equations, drawing lines through the formulas of substances that do not remain as permanent products of the reaction.

$$KI + H_2SO_4 \longrightarrow ____ + ____$$

$$MnO_2 + H_2SO_4 \longrightarrow ____ + ____ + ____$$

Write another equation to show how the two substances that you have crossed out react with each other.

Show the final products of the reaction by completing the following equation:

$$2KI + MnO_2 + 2H_2SO_4 \longrightarrow ____ + ____ + ____ + I_2$$

(*b*) **Effect of various solvents on iodine.**

With a splinter remove small portions of the solid that adheres to the sides of the tube, and try to dissolve the iodine in (1) water, (2) alcohol, (3) a water solution of potassium iodide, (4) carbon disulphide (or chloroform).

Record your results in a tabular form:

TABLE

Solvent	Color of Solution	Degree of Solubility (Slightly, moderately, or very)

(*c*) **Carbon disulphide test for iodine.**

Put 3 cc. of water and a very small crystal of iodine into a test tube; into another test tube put an equal volume of a solution of potassium iodide. To each test tube, add a few drops of carbon disulphide (or chloroform). Shake each mixture thoroughly.

In which case does the carbon disulphide acquire a color?

Second

What is the color?

In what state must the iodine be to give the test with carbon disulphide?

KI

(*d*) **Replacement of iodine by other halogens.**

To about 3 cc. of a very dilute solution of potassium iodide, add a few drops of chlorine water.

Result?

Add chloroform (or carbon disulphide) and shake the mixture.

Result ?

Complete the equation:

$$2\,KI + Cl_2 \longrightarrow \quad + \quad$$

To another portion of potassium iodide solution, add bromine water and chloroform. Shake the mixture.

Result ?

Complete the equation:

$$2\,KI + Br_2 \longrightarrow \quad + \quad$$

Under what conditions does one element replace another ? (*See Experiment 27 and the table it contains.*)

Which of the halogens gives the greatest heat of formation in forming compounds with a given element ?

Which gives least ?

Which of the halogens is most easily replaced by other members of the family ?

Which is least easily replaced ?

Arrange the halogens in the order of their replacing power.

EXPERIMENT 48

The Halogen Acids

APPARATUS. Three test tubes; test tube rack; bunsen burner.

MATERIAL. Sodium chloride; sodium or potassium bromide; sodium or potassium iodide; concentrated sulphuric acid; blue litmus paper.

(*a*) To 1 gram sodium chloride in a test tube, add a few drops of concentrated sulphuric acid. Warm gently.

Result?

Bring a strip of moist litmus to the mouth of the test tube.

Result?

Blow across the mouth of the test tube and notice the fuming of the gas with the moisture of the breath. The amount of the fuming roughly indicates the quantity of the acid issuing from the tube.

If hydrogen chloride were unstable, into what two elements would it decompose?

What would be the color of such a mixture of gases?

Do you find this color in the test tube in which you generated the hydrochloric acid?

What is your decision as to the stability of this acid?

Write the equation for its preparation.

$2NaCl + H_2SO_4 \rightarrow Na_2SO_4 + 2HCl$

(*b*) To 1 gram of potassium bromide in a test tube, add a few drops of concentrated sulphuric acid. Warm gently, if necessary.

Result?

Test the gas with moist litmus.

Result ?

Complete the equation:

$$NaBr + H_2SO_4 \longrightarrow Na_2SO_4 + ___$$

What is the color of the gas in the test tube ?

What element gives this color and what does it indicate as to the stability of the hydrobromic acid ?

Smell the gas *very cautiously.*

Can you detect the odor of sulphur dioxide ?

Of what acid is sulphur dioxide the anhydride ?

What name is given to the process by which sulphuric acid is converted into sulphurous acid ?

Complete the equation:

$$H_2SO_4 + 2\,HBr \longrightarrow H_2SO_3 + ___ + ___$$
$$\longrightarrow ___ + SO_2 + ___ + ___$$

Where did the free bromine come from in this experiment ?

(*c*) To 1 gram of potassium iodide in a test tube add a few drops of concentrated sulphuric acid.

Test the gas with litmus and determine amount of fuming, as before.

Results ?

What do these results show about the amount of hydriodic acid issuing from the tube ?

Compare the stability of this acid with that of hydrobromic acid.

Smell the gas cautiously.

Result ?

What process would form hydrogen sulphide from sulphuric acid ?

State how this is accomplished by the hydriodic acid, accounting for the production of the iodine at the same time. (Compare with action of sulphuric acid on potassium bromide.)

Complete the equations:

$$8\,NaI + \quad H_2SO_4 \longrightarrow \quad Na_2SO_4 + \quad$$

$$\quad H_2SO_4 + 8\,HI \longrightarrow H_2S + \quad + \quad$$

Which of these three halogen acids is most stable ?

Which has the greatest heat of formation (see table below) ?

Which is the most easily oxidized by sulphuric acid? Give a reason for your last answer.

HEATS OF FORMATION OF THE HALOGEN ACIDS

Hydrochloric acid	+ 22,000 calories
Hydrobromic acid	+ 8,400 calories
Hydriodic acid	− 7,000 calories

EXPERIMENT 49

Destructive Distillation

APPARATUS. Hard glass test tube; two sets of stoppers and delivery tubes, as shown (Figure 41); two 8 in. test tubes; ring-stand; clamp; bunsen burner.

MATERIAL. Wood (splinters); soft coal; litmus paper.

(*a*) Destructive distillation of wood.

Arrange apparatus as shown (Figure 41), having the lower end of the delivery tube half an inch from the bottom of the condensing tube. Fill a hard glass test tube with splinters of wood. Heat, gently at first and then strongly, until no further change can be noted.

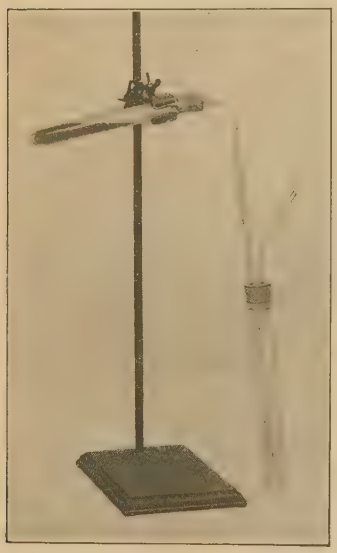


Figure 41.

Describe the appearance of the volatile matter passing off from the wood.

While heating, bring a flame to the end of the jet tube.

Result?

When the action is complete, allow the apparatus to cool, then examine the contents of the test tube.

What is the substance found there?

How does it differ from the original wood?

What is the appearance of the substance in the condensing tube?

Describe any distinct layers that can be distinguished.

Test the liquid in the condensing tube with litmus paper.

Result?

Note the odor of the liquid.

This tarry distillate is a mixture called pyroligneous acid. It contains wood alcohol, acetic acid, acetone, tar, and other substances.

The decomposition by means of heat of a complex substance such as wood, into simpler substances, some of which are condensed to liquids, is called *destructive distillation.*

Name three direct products of the destructive distillation of wood.

(*b*) **Destructive distillation of coal.**

Free the hard glass tube from charcoal, and half fill it with finely crushed soft coal. Replace the condenser and tubes with a fresh set, and heat as before.

Describe the appearance of the volatile matter in this case.

Is it the same as that obtained from the wood?

While the heating is going on, lay a piece of red litmus paper over the end of the jet tube.

Result?

What kind of a compound is shown to be present by this test?

Bring a flame to the end of the jet tube.

Result?

Heat the tube until gas is no longer given off. Then allow the tube to cool. When you can handle the hard glass tube, pour its solid contents on the base of the ring-stand.

Describe the residue as to color, structure, and weight, as compared with the original coal.

This residue is coke.

Examine the condensing tube.

Describe the liquid deposited there.

Why is the distillation of soft coal a destructive distillation?

Name three direct products of this destructive distillation.

DRAWING

EXPERIMENT 50

Properties of Carbon

APPARATUS. Hard glass test tube, 6″, with one-hole stopper and bent delivery tube; two test tubes; beaker, 200 cc.; evaporating dish; funnel; ring-stand with clamp, and one ring to support the funnel; stirring rod; bunsen burner; small sheet of paper.

MATERIAL. Copper oxide, powdered; charcoal, powdered; limewater; boneblack; sugar; copper sulphate solution, 1 to 40; filter paper; cider vinegar.

(*a*) Carbon as a reducing agent.

Arrange the apparatus as shown in Figure 42. Fill one-tenth of a test tube with copper oxide, then pour the oxide on a sheet of paper. Using the same test tube, measure an equal volume of powdered charcoal. Add the charcoal to the copper oxide on the piece of paper, and mix the two thoroughly. Pour the mixture into the hard glass test tube shown in the figure. Pour limewater into the test tube into which the delivery tube extends until the limewater just touches the end of the delivery tube.

Figure 42.

Heat the hard glass test tube, cautiously at first, commencing at the part around the upper portion of the mixture, and gradually moving the flame toward the closed end of the tube.

What change do you observe in the limewater at first?

Carbon dioxide is the anhydride of carbonic acid.

Complete the equation:

$$CO_2 + H_2O \longrightarrow H_2CO_3$$

What base is in solution in limewater?

Write the equation for the neutralization of this base with carbonic acid.

Allow the tube to cool, and when cold pour its contents into a 200 cc. beaker. Let a small stream of water flow into the beaker.

What substance is carried away by the water?

What is left in the beaker?

What element is taken from the copper oxide?

Complete the equation:

$$2\,CuO + C \longrightarrow 2\,Cu + CO_2$$

What name is given to the process of removing oxygen from a compound?

What kind of an agent is carbon in this case?

(*b*) **Carbon as a decolorizer.**

Thoroughly mix a little boneblack with some cider vinegar in an evaporating dish. Fold a sheet of filter paper and place it in the funnel. Pour boneblack on the filter and scoop out a hollow in the center of the boneblack. Into this hollow, pour the mixture of vinegar and boneblack. Collect the filtrate and note its color.

Result?

Put enough sugar into a test tube to fill the rounded part. Heat this slowly and evenly, rotating the tube as the sugar melts. The sugar should be well browned, but not burned. The product contains caramel, a substance used as a flavor in

cooking. As soon as the tube containing the caramel has cooled, fill two thirds of it with water, and warm the mixture until the solid has dissolved. The solution thus obtained is used to illustrate the impure sugar solution of a sugar refinery. The heat used in obtaining crude sugar helps to give the product a brown color which must be removed to get white sugar.

Filter the solution of sugar and caramel as you did the cider vinegar.

Result? colorless

Filter a dilute solution of copper sulphate through boneblack.

Result? just as blue as before

Can the color be removed from all liquids by filtering them through boneblack?

no

DRAWING

EXPERIMENT 51

Preparation and Properties of Carbon Dioxide

APPARATUS. Wide-mouth bottle, 8 oz., with two-hole rubber stopper to fit, carrying thistle tube and delivery tube; three wide-mouth bottles; three test tubes; glass tube; enamelled ware dish or pneumatic trough; three glass plates.

MATERIAL. Marble chips; concentrated hydrochloric acid; limewater; blue litmus solution; candle; wood splinter.

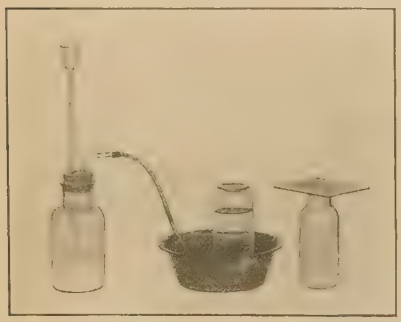


Figure 43.

(*a*) **Preparation.**

Into a bottle provided with a delivery tube and a thistle tube reaching nearly to the bottom of the bottle, put marble chips to about the depth of an inch. Cover the marble with water, and add concentrated hydrochloric acid, a few cubic centimeters at a time, so as to get a moderate action. Collect three bottles of the gas by the displacement of water (Figure 43) for use in parts (*a*), (*b*), (*d*), and (*e*).

Describe the action in the generator.

Complete the equation:

$$CaCO_3 + 2HCl \longrightarrow H_2CO_3 + \underline{\qquad}$$

Write the equation for the decomposition of carbonic acid.

Represent the entire action by one equation.

Is carbonic acid stable at ordinary temperatures?

Why can any of the common acids be used in preparing carbon dioxide?

(*b*) **Odor and color.**

Inhale some of the gas from a bottle.

What is the effect of the gas on the nose?

Has carbon dioxide color?

(*c*) **Solubility.**

Let the gas from the generator bubble through a test tube half full of blue litmus solution.

What change takes place?

(If the result is not evident to you, compare the color of the liquid in the test tube with that of the litmus solution in the stock bottle.)

Why is carbon dioxide called an acid anhydride?

Write the equation for the reaction of carbon dioxide with water.

$CO_2 + H_2O \rightarrow H_2CO_3$

What does the method of collection show in regard to the extent to which carbon dioxide is soluble in water?

How is the solubility of the gas increased in the preparation of effervescent drinks? (*Class discussion.*)

(*d*) **Relative weight and relation to combustion.**

Slowly invert a bottle of carbon dioxide over a lighted candle.

Results?

Candle goes out

What three properties of carbon dioxide are shown?

Heavier than air.

not combustible

(*e*) **Action with limewater.**

Pour half a test-tubeful of limewater into a bottle of carbon dioxide and shake the bottle.

Result?

Write the equation to show the formation of the precipitate, calcium carbonate.

(*f*) **Product of combustion.**

Burn a wooden splinter in a bottle of air. Add limewater and shake the bottle.

Result?

precipitate

How does this show that the splinter contains carbon?

Blow through a glass tube into a test tube half full of limewater.

Result?

precipitate

What gas do we exhale?

carbon dioxide

How is it produced in the body?

going on in Body,

EXPERIMENT 52

Chemical Fire Extinguisher

APPARATUS. Four test tubes, 6″; one-hole rubber stopper to fit test tubes and carrying a delivery tube; wide-mouth bottle, 6 oz.; test tube, 4″ × ½″; one-hole rubber stopper to fit 6 oz. bottle; glass tube 4″ long; glass tube 2″ long drawn out so as to deliver a stream 2 mm. in diameter; 2′ of rubber tubing to fit glass tubes; bunsen burner; ring-stand with small clamp; pan.

MATERIAL. Limewater; sodium bicarbonate; sulphuric acid, 1 to 10; splinters of wood.

(*a*) Pour 15 cc. of limewater into a test tube and set it aside for future use. Close the mouth of another test tube with a one-hole rubber stopper carrying a delivery tube. Arrange the apparatus so that the delivery tube will dip into the limewater in the first test tube.

Pour into the test tube generator enough sodium bicarbonate to fill the curved portion of the test tube; add about 10 cc. of water and a few drops of sulphuric acid, then immediately replace the stopper.

What is the name of the gas evolved?

Complete the equation:

$$2\,NaHCO_3 + H_2SO \longrightarrow _____ + _____ + _____$$

(*b*) Arrange the apparatus as in part (*a*), using a dry test tube for the generator. Pour into the test tube generator about 5 cc. of sodium bicarbonate and heat it until the flame in contact with the test tube turns yellow. This indicates that the glass is beginning to soften. Continued heating would melt the glass.

What change takes place in the limewater?

What collects on the cool portion of the tube?

What are two of the substances produced by the decomposition of sodium bicarbonate?

Complete the equation:

$$2\,NaHCO_3 \longrightarrow NaCO_3 + _____ + _____$$

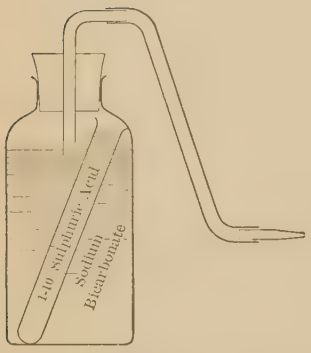


Figure 44.

Why is sodium bicarbonate the chief ingredient of dry-powder fire extinguishers?

(*c*) Arrange apparatus as shown in Figure 44. Pour a nearly saturated solution of sodium bicarbonate into the wide-mouth bottle until the surface of the liquid reaches the curved portion of the bottle near its neck. Fill the small test tube with very dilute sulphuric acid (1 to 10) and stand the test tube in the bottle. Replace the stopper and set the bottle on the table.

Figure 45.

Build a small fire of splinters of wood in a pan placed in a sink, or wherever the instructor may direct. Hold the stopper of the fire extinguisher *firmly pressed* into the mouth of the bottle (Figure 45); point the end of the delivery tube at the fire, and invert the bottle. Put out the fire.

What gas was generated by the reaction between the sodium bicarbonate and the sulphuric acid?

Why was the liquid forced out of the bottle?

What two advantages are there in using more than enough sodium bicarbonate to neutralize the sulphuric acid?

EXPERIMENT 53

Hard Waters

APPARATUS. Carbon dioxide generator, consisting of wide-mouth bottle with two-hole stopper to fit, carrying thistle tube and delivery tube for leading gas to the bottom of a test tube; four test tubes; small funnel; stirring rod; bunsen burner.

MATERIAL. Calcium sulphate (plaster of Paris); magnesium sulphate; marble chips; dilute hydrochloric acid, 1 : 4; limewater; distilled water; filtered soap solution; filter paper.

(*a*) Add a drop or two of soap solution to distilled water in a test tube and shake the tube.

Are lasting suds produced?

This result is characteristic of "soft" waters.

Drop a pinch of magnesium sulphate into a test tube two thirds full of water. Close the mouth of the tube with the thumb and shake it thoroughly. Add a few drops of the soap solution. Shake the tube.

Do suds form?
What is produced?

This is an insoluble magnesium soap. Waters that behave in this way with soap solution are "hard" waters.

Continue the addition of soap solution until suds finally form.

Why does it cost more to wash with hard water than with soft water?

What would tend to become entangled in the fibers of a fabric when clothes are washed with hard water?

(*b*) Put some pieces of marble into a generator and cover them with dilute hydrochloric acid. Lead the carbon dioxide

formed to the bottom of a test tube two thirds full of lime-water.

What is the first result?

The insoluble compound formed is calcium carbonate.

Write the equation for its precipitation.

What happens to the precipitate on the continued passing of the carbon dioxide?

The compound formed is calcium bicarbonate, $CaH_2(CO_3)_2$.

Is it soluble or insoluble?

What acid was formed when the excess of carbon dioxide reacted with the water in the test tube?

What effect did this acid have on the precipitate, calcium carbonate?

Complete the equation:

$$CaCO_3 + ___ + ___ \longrightarrow ___$$

(*c*) Pour a little of the water solution of calcium bicarbonate into another test tube. To this portion, add a few drops of soap solution and shake the tube.

What kind of water is the water containing calcium bicarbonate in solution?

In a separate test tube heat gently another portion of the calcium bicarbonate solution.

What happens on warming the liquid, particularly near the walls of the test tube?

Hold a stirring rod with a drop of limewater on its end above the heated liquid in the test tube.

What is the effect on the limewater?

What does this show?

Continue heating the liquid in the test tube until it boils.

What is the second effect of heat on a water solution of calcium bicarbonate?

Filter the contents of the tube just heated. Test the filtrate with a few drops of soap solution.

Has the water solution of calcium bicarbonate been softened? Explain.

Such a hard water is called a "temporary" hard water.

Complete the equation for the softening of a temporary hard water by boiling:

$$CaH_2(CO_3)_2 \longrightarrow CaCO_3 + \underline{\quad} + \underline{\quad}$$

Account for the crust formed on the inside of tea kettles in which temporary hard water is boiled day after day.

(*d*) Add a pinch of calcium sulphate (plaster of Paris) to a test tube two thirds full of water. Shake the tube, filter the contents, and divide the filtrate into two portions. Using one, determine with soap solution whether or not the water is hard.

Result?

Boil the second portion, and then test with soap solution.

Result?

Is the water solution of calcium sulphate softened by boiling?

Such water is "permanent" hard water.

Would permanent hard water form a deposit on a teakettle?

Explain.

A water often contains both temporary and permanent hardness.

How could you show the presence of each kind of hardness in the presence of the other?

EXPERIMENT 54

Baking Powders

APPARATUS. Four test tubes; rubber stopper, one-hole, to fit test tube and carrying a delivery tube as shown in Figure 46; test tube rack.

MATERIAL. Sodium bicarbonate; limewater; hydrochloric acid, 1 to 4; potassium acid tartrate; monocalcium phosphate; [redacted] phosphate; sodium alum; blue litmus paper.

Figure 46.

(*a*) Reaction between sodium bicarbonate and acids.

Arrange apparatus as shown in Figure 46. Pour about 10 cc. of limewater into the test tube into which leads the long arm of the delivery tube. Remove the stopper and put a pinch of sodium bicarbonate (baking soda) in the tube; add a few drops of dilute hydrochloric acid and immediately replace the stopper.

What gas is liberated by the reaction?

carbon dioxide

Write the equation representing the reaction.

$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$

Sour milk contains lactic acid, $H(C_3H_5O_3)$.

Why is a mixture of sour milk and baking soda used as a leavening agent?

Forms carbon dioxide neutralizes

Equation?

$HC_3H_5O_3 + NaHCO_3 \rightarrow NaC_3H_5O_3 + H_2O + CO_2$

(*b*) Cream of tartar baking powders.

Sprinkle a few particles of cream of tartar, potassium acid tartrate, $KH(C_4H_4O_6)$, on a dry piece of blue litmus paper.

Result?

Wet the powder and paper.

Result?

Into what ions would potassium hydrogen tartrate be dissociated by water?

Equation?

What kind of ions produced the action on the litmus paper?

Mix a little dry cream of tartar with a little dry baking soda, and pour the mixture into a dry test tube arranged with a one-hole stopper carrying a delivery tube that extends into some limewater in another test tube. Add a little water to the mixture of cream of tartar and baking soda, and immediately replace the stopper carrying the delivery tube.

Result?

Account for the fact that carbon dioxide is not evolved from a dry mixture of sodium bicarbonate and potassium acid tartrate, but is generated as soon as the mixture is wet.

Complete the following equation, representing the reaction that takes place in the presence of water:

$$NaHCO_3 + KH(C_4H_4O_6) \longrightarrow NaK(C_4H_4O_6) + ____ + ____$$

Cream of tartar baking powders consist of a mixture of cream of tartar, sodium bicarbonate, and some inert substance, such as starch or flour, which is used as a filler.

Calculate the number of grams of sodium bicarbonate that would react with 10 grams of potassium acid tartrate.

What volume of carbon dioxide, measured at standard conditions, would be evolved when the mixture was wet?

What volume would the gas occupy at 21° C. and a pressure of 770 mm.?

One of the cream of tartar baking powders is prepared by mixing dry 2 parts by weight of cream of tartar, 1 part of sodium bicarbonate, and 1 part of starch.

(*c*) **Phosphate powders.**

Sprinkle a few particles of monocalcium phosphate, $CaH_4(PO_4)_2$ on a wet piece of blue litmus paper.

Result?

What ion derived from the monocalcium phosphate produces this change?

Repeat the experiment, using monosodium phosphate, NaH_2PO_4, in place of monocalcium phosphate.

Account for the reaction.

Some phosphate baking powders consist of dry mixtures of monocalcium phosphate, sodium bicarbonate, and a filler; other phosphate baking powders are dry mixtures of monosodium phosphate, sodium bicarbonate, and a filler.

(*d*) **Alum baking powders.**

Sprinkle a few particles of sodium alum, ~~sodium~~ aluminum ~~sulphate,~~ $NaAl(SO_4)_2$ on a wet piece of blue litmus paper.

Result?

Sodium aluminum sulphate is a double salt, consisting of sodium sulphate in combination with aluminum sulphate. Aluminum sulphate is hydrolyzed in solution (see § 183, First Principles of Chemistry).

Write an equation to show this hydrolysis.

Why does a water solution of alum give an acid reaction?

Alum baking powders consist of dry mixtures of alum, sodium bicarbonate, and a filler.

Why should a baking powder be stored in an air-tight container?

Should a baking powder be mixed with the flour before or after the addition of water?

Why?

EXPERIMENT 55

Preparation and Properties of Carbon Monoxide

APPARATUS. Ring-stand with 1 ring and 1 clamp; wire gauze with asbestos center; beaker, 200 cc.; side arm test tube, with single hole rubber stopper to fit; small thistle tube; delivery tube; enameled pan; two wide-mouth bottles, 4 oz.; glass plate; bunsen burner.

MATERIAL. Formic acid, sp. gr. 1.2; concentrated sulphuric acid; limewater.

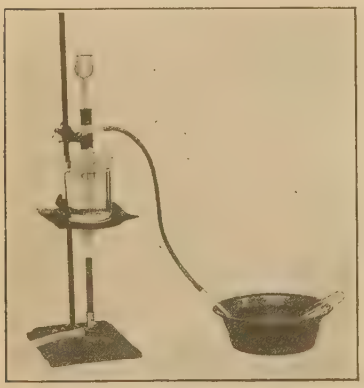


Figure 47.

(*a*) **Preparation.**

Arrange the apparatus as shown in Figure 47. Pour 5 cc. of formic acid and an equal amount of concentrated sulphuric acid through the thistle tube into the side arm test tube. Heat the water in the beaker until it boils. Collect the gas in small bottles as long as it is evolved. **Do not let carbon monoxide escape into the room; it is very poisonous.** Get rid of the gas in any of the bottles that you do not need by burning it.

(*b*) **Properties.**

Burn a bottle of carbon monoxide.

Describe the color of the flame.

Compare this flame with that of burning hydrogen.

Pour 15 cc. of limewater into the bottle in which the carbon monoxide has been burned and shake the liquid around gently.

Result?

What gas is formed by the burning of carbon monoxide?

Write the equation for this reaction.

(*c*) **General questions.**

The formula of formic acid is $HCHO_2$.

Write the equation for its decomposition.

Explain how concentrated sulphuric acid helps to bring about this decomposition.

EXPERIMENT 56

Preparation and Properties of Carbon Monoxide

Second Method

APPARATUS. Flask, 250 cc.; two-hole rubber stopper to fit flask, provided with thistle tube; delivery and connecting tubes as shown in Figure 48; two wide-mouth bottles, 6 oz., provided with two-hole rubber stoppers and tubes as shown in the figure; three wide-mouth bottles, 6 oz.; pneumatic trough; glass plate; ring-stand with 1 ring and 1 clamp; wire gauze with asbestos center; bunsen burner; two test tubes; single hole rubber stopper to fit test tube, provided with L delivery tube.

MATERIAL. Crystallized oxalic acid; concentrated sulphuric acid; caustic soda solution, 300 g. to the liter; limewater.

Caution! Concentrated sulphuric acid, especially when hot, produces extremely serious burns; also carbon monoxide is very poisonous.

(*a*) Preliminary test.

Put a pinch of oxalic acid in a test tube and add 1 cc. of concentrated sulphuric acid. Fit the test tube with a rubber stopper and delivery tube. Into a second test tube put 10 cc. of limewater. Heat the contents of the first tube gently, and allow the gas that is given off to pass through the limewater in the second tube.

Result?

What gas is shown as one of the products of the action between oxalic and sulphuric acids?

(*b*) Preparation.

Arrange the apparatus shown in Figure 48. Into the flask put 15 grams of oxalic acid. Into each of the two wide-mouth bottles put 30 cc. of caustic soda solution. Make sure that the tube by which the gas enters a bottle reaches nearly to the

bottom, and that the tube by which the gas leaves reaches only just below the rubber stopper. The thistle tube should reach nearly to the bottom of the flask.

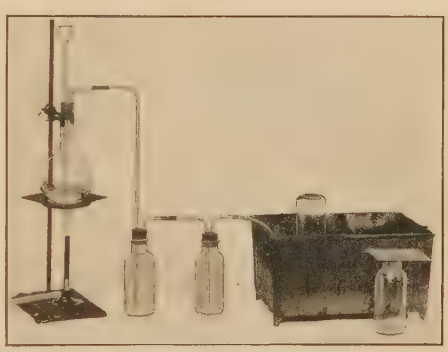


Figure 48.

Pour about 20 or 30 cc. of sulphuric acid through the thistle tube into the flask. Heat the mixture with a small flame. When the action has started, remove the flame, but replace if the action becomes too slow. Collect in bottles in the pneumatic trough the gas that is given off.

Compare the rates at which bubbles pass through the two wash bottles.

Why are two bottles of caustic soda solution necessary?

(*c*) **Properties.**

As soon as a bottle fills with the gas, remove it from the trough and hold its mouth to the flame.

Why did the first bottles fail to ignite?

Repeat the experiment, using a second bottle of the gas.

Describe a carbon monoxide flame as to brightness and color.

When you have burned a bottle of carbon monoxide, pour into it 20 cc. of limewater. Shake the limewater around in the bottle gently for a moment.

Result?

What is the product formed when carbon monoxide burns?

Write the equation for the reaction.

(*d*) **General questions.**

What two gases result from the decomposition of oxalic acid by the aid of sulphuric acid?

From an inspection of the formula of oxalic acid, $H_2C_2O_4$, *decide what a third product is.*

Explain how sulphuric acid produces the action.

Write the equation for the decomposition of oxalic acid.

What happens in the wash bottles to the gas that was shown to be present in part (*a*)*?*

Write an equation to show this.

Why might the flames of hydrogen and of carbon monoxide be confused?

How could you distinguish them by a test of the products of combustion?

EXPERIMENT 57

Borax and Boric Acid

APPARATUS. Beaker, 100 cc.; stirring rod; funnel; graduate, 50 cc.; evaporating dish; ring-stand with two rings; wire gauze with asbestos center; bunsen burner; balance with weights; pan of cold water; test tube.

MATERIAL. Filter paper; borax; sulphuric acid, concentrated; alcohol; hydrochloric acid, 1 to 4; ammonia water, concentrated; turmeric paper.

(*a*) Preparation of boric acid.

Weigh out, on a balanced filter, 12 grams of borax. Pour 50 cc. of water into a beaker and heat it to boiling on a wire gauze placed over a bunsen flame. Dissolve the borax in the hot water. Measure out 6 cc. of sulphuric acid and *very slowly* pour it down a stirring rod held just above the solution of borax, so that the acid will enter the solution *drop by drop*.

Cool the solution by setting the beaker in a pan of cold water. As the cooling proceeds, boric acid will crystallize out. Filter the mixture and wash the boric acid on the filter three or four times. Use a few cubic centimeters of *cold* water at a time and allow one portion of water to run through the filter before adding a second portion.

What is the formula for the acid of which borax, $Na_2B_4O_7$, *is a salt?*

This acid combines with water to form boric acid, H_3BO_3.

Taking these facts into consideration, write equations representing in two steps, the production of boric acid by the method you have employed. Then represent the reactions by a single equation.

Account for the fact that the reaction runs in one direction (*see Experiment* 28).

Could hydrochloric acid be used in place of sulphuric acid?

Why do you think so?

What by-product was left in the liquid from which the boric acid crystallized?

Why were better results obtained by repeatedly washing the boric acid than could have been obtained by using the same quantity of wash water at one time?

(*b*) **Alcohol test for boric acid.**

Place some of the boric acid you have prepared in an evaporating dish and *just cover* it with alcohol. Set fire to the alcohol.

What color of the flame in addition to yellow do you observe?

Treat a little borax in the same manner that you have just treated the boric acid.

How does this flame differ in color from the preceding flame?

(*c*) **Turmeric test for boric acid.**

Dip a piece of turmeric paper in a solution of boric acid and then dry the paper by wrapping it around a test tube containing boiling water.

What change in the color of the turmeric do you observe?

Add to the paper a little ammonium hydroxide.

What change in color do you observe?

Determine whether dilute hydrochloric acid will restore the color of turmeric paper after it has been changed by boric acid and ammonium hydroxide.

Result?

(*d*) **Test for borax.**

Acidulate a solution of borax with hydrochloric acid and make the turmeric paper test for boric acid.

EXPERIMENT 58

Water Softening

APPARATUS. Dropping bottle with notched cork; funnel; four test tubes; bunsen burner.

MATERIAL. Calcium sulphate (plaster of Paris); magnesium sulphate; temporary hard water; limewater; sodium carbonate solution; filtered soap solution; filter paper.

(*a*) Boil one third of a test tube of temporary hard water.

Result ?

How does the boiling soften the water?

Complete the equation:

$$CaH_2(CO_3)_2 \longrightarrow _____ + _____ + _____$$

What becomes of the substance that causes the hardness?

Temporary hard waters, before being fed to boilers, are sometimes brought to boiling in a feed water heater.

Why ?

Why is the water then filtered before passing it into the boiler where steam is generated?

(*b*) To one third of a test tube of temporary hard water add an equal volume of limewater.

Result ?

Write the equation for the action of limewater on the temporary hard water.

What acid must be in excess when calcium bicarbonate is formed?

Write the equation for the neutralization of this acid with lime-water.

Which product is the precipitate?

How is limewater made?

Why is lime added to tanks of temporary hard water in a water-softening plant?

(*c*) Prepare a permanent hard water by shaking a pinch of calcium sulphate (plaster of Paris) in a test tube of water and then filtering the liquid. Pour half of this hard water into another test tube and set it aside for later use.

To the other half of the hard water, slowly add a sodium carbonate solution from a dropping bottle, as long as any precipitate forms. Shake the tube after each addition of the carbonate.

Write the equation for the reaction.

Which of the two products do you know to be an insoluble substance?

Remove the precipitate from the liquid by filtering. Test the filtrate with soap solution to determine the readiness with which it forms permanent suds.

Result?

Similarly try the soap solution with the portion of the original hard water that was set aside.

Which forms suds more readily, the original hard water, or the water to which sodium carbonate was added?

(*d*) Take half a test tube of temporary hard water and add to it a pinch of calcium sulphate and a pinch of magnesium sulphate. Add water to fill the test tube and mix the contents by shaking. If the liquid is not clear, filter it. This represents a common type of hard water that contains several causes of hardness.

To the hard water add limewater, a few drops at a time, as long as a precipitate forms. Filter the liquid. To the filtrate add sodium carbonate solution from the dropping bottle as long as precipitation occurs.

Filter the liquid and test the filtrate with soap solution.

Result ?

Was the hardness due to magnesium removed from the water by the lime and soda treatment (i.e. limewater and sodium carbonate)?

EXPERIMENT 59

Bleaching of Cotton

APPARATUS. Porcelain mortar and pestle; 1000 cc. graduate; two beakers, 250 cc.; glass stirring rods; ring-stand with ring; wire gauze with asbestos center; bunsen burner.

MATERIAL. Unbleached cotton waste; litmus paper; solutions prepared as directed below; bleaching powder; sodium hydroxide; sodium thiosulphate; concentrated sulphuric acid; starch; sodium iodide.

Preparation of Solutions. The quantities given are sufficient for a class of ten pupils.

Alkali. Dissolve 16 g. of sodium hydroxide in 1000 cc. of water.

Bleaching Solution. Work 10 g. of a good quality of bleaching powder into a smooth, thin paste with a little water in a porcelain mortar. Rinse the paste into a 1000 cc. graduate, and then add water until the graduate is filled to the 800 cc. mark. Pour the mixture into a bottle, shake it thoroughly, and then allow the undissolved matter to settle. Use only the clear supernatant liquid.

Acid. Pour 30 cc. of concentrated sulphuric acid into 500 cc. of water.

Antichlor. Dissolve 2 g. of sodium thiosulphate ("hypo") in 500 cc. of water.

Starch Iodide Paste. Mix some starch with cold water and slowly pour the mixture into 50 cc. of boiling water. Stir constantly and discontinue the addition of starch and the boiling as soon as a thin paste has been obtained. Add 5 cc. of a dilute solution of sodium iodide to the starch paste and mix the two thoroughly by stirring.

(*a*) Cotton yarn and cloth are "boiled out" to remove wax, grease, and dirt which would prevent the uniform action of the bleach on the fibre.

Pour 100 cc. of the alkali solution into a 200 cc. beaker and immerse in the liquid about a gram of cotton waste. Heat the liquid to boiling, and continue to boil it gently for 10 minutes, meanwhile keep the cotton in motion in the liquid by means of a stirring rod.

What evidence is there that substances have been removed from the cotton?

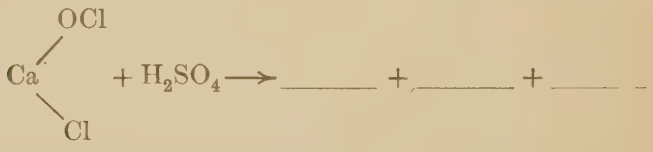
Remove the waste and rinse it repeatedly in water.

(*b*) Pour 50 cc. of the bleaching solution into a beaker and put the wet cotton waste into it. By means of a glass rod, work the cotton in the liquid for at least five minutes and then allow it to remain in the liquid another five minutes.

(*c*) Remove the waste from the bleaching solution and, without rinsing it, put it into a beaker containing 50 cc. of the acid and work it in the liquid for five minutes.

Acids react with bleaching powder to produce a calcium salt of the acid, water, and chlorine.

Complete the equation:



Chlorine reacts with water to produce hydrochloric acid and oxygen, which changes the coloring matter of many colored compounds into a colorless substance.

(*d*) **Use of antichlor.**

Free chlorine must not be permitted to remain in contact with the cotton fiber, because chlorine weakens the strength of the fiber by reacting with it.

Wash the cotton to remove the acid.

How can you determine when this has been accomplished?

Put a little of the starch iodide paste on the cotton. Look carefully to see whether a blue tint is developed. Chlorine liberates iodine from sodium iodide, and iodine reacts with starch to yield a blue substance.

Complete the equation:

$$Cl_2 + NaI \longrightarrow _____ + _____$$

Soak the cotton waste in 50 cc. of the antichlor, rinse the cotton thoroughly, and again use the starch iodide paste to test for chlorine.

Balance the equation:

$$Na_2S_2O_3 + H_2O + Cl_2 \longrightarrow H_2SO_4 + HCl + Na_2SO_4.$$

EXPERIMENT 60

Four Ways of Preparing a Salt, Sodium Chloride

These methods are placed together for convenience. The exercise should be assigned in advance, and the calculations in part (*c*) should be made before coming to the laboratory.

APPARATUS. Apparatus for making chlorine, 1 set for each group of five pupils. Apparatus similar to that shown in Figure 18 is convenient, and consists of a 250 cc. flask with 2-hole stopper carrying a thistle tube and a delivery tube arranged for use in the collection of a gas by the downward displacement of air, a ring-stand with 1 ring, a clamp, a small pan of water, and a bunsen burner. Bottle, 8 oz.; cover glass; ring-stand with one ring; bunsen burner; bottle with notched stopper for dropping hydrochloric acid; two test tubes; funnel; evaporating dish; stirring rod; deflagration spoon; graduate, 100 cc.; wire gauze with asbestos center.

MATERIAL. Fine sand; sodium; manganese dioxide; hydrochloric acid, concentrated; hydrochloric acid, 1 to 4; sodium hydroxide solution, 1 to 10; solution of crystallized barium chloride, 104 grams of $BaCl_2 \cdot 2\,H_2O$ per liter; solution of crystallized sodium sulphate, 137.5 grams of $Na_2SO_4 \cdot 10\,H_2O$ per liter; sodium carbonate, dry; litmus paper, red and blue.

(*a*) Direct combination.

This part of the experiment should be performed under a hood.

Collect an 8 oz. bottle of chlorine prepared by any convenient method, for example that described in Experiment 18.

Half fill the bowl of a deflagration spoon with sand. Ask the instructor to place a piece of clean sodium on the sand. At once take the sodium to the hood containing the bottle of chlorine, ignite the sodium by directing a flame against it, and immediately lower the burning sodium into the chlorine.

What chemical reaction takes place between the sodium and the chlorine?

Why did it occur readily (see Experiment 26)?

By what properties can you identify the product?

Name the product.

(*b*) **Neutralization.**

In a porcelain evaporating dish, neutralize 5 cc. of a solution of sodium hydroxide with a solution of dilute hydrochloric acid. Evaporate the neutral solution to dryness.

What is the name of the solid obtained?

What ion does every water solution of an acid contain?

What ion does every water solution of a base contain?

What becomes of these ions during the process of neutralization?

What ions are left in solution after the sodium hydroxide solution is neutralized with hydrochloric acid?

What compound is formed on evaporation by the combination of these ions?

Why do neutralization reactions tend to go to an end?

(*c*) **Double replacement due to an insoluble product.**

Write the equation for the reaction between a barium chloride solution and a solution of sodium sulphate.

Why does the reaction go to an end?

Using the equation

$$Na_2SO_4 \cdot 10\ H_2O + BaCl_2 \cdot 2\ H_2O \rightarrow BaSO_4 + 2\ NaCl + 12\ H_2O,$$

and the table of atomic weights on page 234, calculate

(*a*) *the number of grams of crystallized sodium sulphate.*

(*b*) *the number of grams of crystallized barium chloride, required to prepare* 1 *gram of sodium chloride.*

The stock solution of barium chloride contains 104 grams of crystallized barium chloride per liter. The stock solution of sodium sulphate contains 137.5 grams of crystallized sodium sulphate per liter.

Calculate the number of cubic centimeters of (*a*) *the stock solution of barium chloride,* (*b*) *the stock solution of sodium sulphate, containing the weights of barium chloride and sodium sulphate respectively that you found would react to yield* 1 *gram of sodium chloride.*

Why are these calculations necessary?

Measure out the volume of barium chloride solution required and pour it into a test tube. Measure out the volume of sodium sulphate required. Pour the sodium sulphate solution into an evaporating dish, heat the solution, and add the barium chloride solution to it. Allow the mixture to stand for a few minutes and then filter it, using a good quality of filter paper. If the first portion of the filtrate is not clear, repeat the operation.

What is the precipitate?

What salt is contained in the filtrate?

Obtain the salt from the filtrate.

How did you accomplish the separation of the two compounds produced?

(*d*) **Double replacement due to the volatility of one of the products.**

Add slowly, with constant stirring, hydrochloric acid, 1 to 4, to about 2 grams of sodium carbonate, until all of the sodium carbonate has dissolved.

What gas is generated?

Account for its production.

Why did the reaction go to an end?

Evaporate the solution to dryness.

What became of the excess of hydrochloric acid?

Why did the reaction go to an end?

EXPERIMENT 61

Cobalt Nitrate Tests

APPARATUS. Plaster block, made by pouring a thin mixture of plaster of Paris and water into a form on a glass or stone slab and cutting the mass just before it hardens into strips of suitable size; blowpipe; forceps; bunsen burner.

MATERIAL. Zinc sulphate; alum; magnesium sulphate; cobalt nitrate solution in bottle provided with dropper; unknowns.

(*a*) Put a little of some zinc compound, as zinc sulphate, in a cavity made in a plaster block with the top of a pair of forceps. Heat it as hot as possible at the end of a small blowpipe flame. Allow the residue to cool.

Record the color of the residue in a table like that on page 171.

Moisten the residue with a drop or two of cobalt nitrate solution. Again heat it intensely, and, on cooling, note the color of the mass that remains on the plaster block.

Record in the table the color obtained.

(*b*) Place an aluminum compound, as alum, in a fresh cavity in the block. Repeat the test made with the zinc compound, so as to obtain a characteristic coloration.

Record the results in the table.

(*c*) Make a fresh cavity in the block, and place in it some magnesium sulphate. Heat the sulphate with the blowpipe until it glows brightly. Cool and moisten the fused mass with *a very little* cobalt nitrate. Blow very vigorously, and allow the mass to cool. Note carefully the delicate coloration.

Record the colors obtained in the table.

(*d*) Obtain from the instructor an unknown compound, and test it with the blowpipe and cobalt nitrate, to determine whether it is a compound of aluminum, magnesium, or zinc.

What color was obtained in the second heating?

What metal did the compound contain ?

TABLE

Compound Taken	Color of Residue after First Heating	Color of Residue after Heating with Cobalt Nitrate

EXPERIMENT 62

Borax Bead Tests

APPARATUS. Mounted platinum wire (or glass rod, 5″ long); bunsen burner; blowpipe, if desired; triangular file.

MATERIAL. Powdered borax; cobalt nitrate or oxide; manganese dioxide; chrome alum, or chromium sulphate; ferric chloride or other iron compound.

(*a*) Bend the end of a platinum wire into the shape of a letter J, 2 mm. across the opening. Heat the loop red-hot and dip it into powdered borax. Heat again in the hottest part of the flame. The borax swells, loses its water of crystallization, and then melts to a transparent glass.

In case a platinum wire is not available, heat the end of a glass rod in the flame. Then dip the hot end into powdered borax. Heat in the hottest part of the flame the borax that sticks to the rod, until the borax swells up to an irregular shaped mass on the end of the rod.

(*b*) Touch the hot irregular mass to a tiny bit of some cobalt compound, as cobalt nitrate. Heat in the hot outer portion of the bunsen flame (oxidizing flame), until a clear glassy bead is obtained. Note the characteristic color.

Record in a table arranged as on page 173.

To remove the bead from the platinum wire, heat it red-hot and quickly shake off the molten bead into the sink or waste jar. Make a fresh bead and examine it. If it is not colorless, repeat the operation.

To remove the bead from the glass rod, scratch it with a sharp triangular file, and break it off as you would cut a piece of glass tubing; or let a drop of water fall on the hot rod and tap the end of the rod on a table.

(*c*) Touch a newly made bead while hot to a bit of man-

ganese dioxide. Fuse as before. Note the characteristic color given by manganese compounds.

Record in the table.

(*d*) Using a chromium compound, *e.g.* chrome alum or chromium sulphate, obtain in a similar manner a characteristic bead.

Record result in the table.

(*e*) Obtain the color characteristic of an iron compound in the oxidizing flame.

Record result in the table.

TABLE

Compound Taken (Name)	Formula	Color of Bead

EXPERIMENT 63

Indentification of Simple Salts

APPARATUS. Platinum wire; cobalt glass; charcoal or plaster block; blowpipe; test tubes; bunsen burner.

MATERIAL. Borax; solutions of cobalt nitrate, silver nitrate, barium chloride, and ferrous sulphate; dilute nitric and hydrochloric acids; concentrated sulphuric acid; unknown.

Use a very small portion of the unknown given you in making each test. Always keep a portion for verification of your results.

Keep a tabulated record of all tests, even those giving negative results.

(*a*) Determine the metallic part of the substance by means of (1) the flame test for sodium and potassium, (2) cobalt nitrate test, (3) borax bead test.

(*b*) Then put a fresh portion of the unknown into a test tube and try to dissolve it in water, heating if necessary. Filter if there is an undissolved residue.

Divide the clear liquid into several portions and make tests for a chloride, a sulphate, and a nitrate.

(*c*) Now determine whether the unknown contains a carbonate by the addition of an acid and making the limewater test of the gas that is given off.

Finally decide what you have found in the unknown substance, with reasons for your decision. Then take your note book to the instructor.

Number of unknown _____ .

TABLE

Test	Result

The unknown contains _____

EXPERIMENT 64

Action of Metals on Salt Solutions

APPARATUS. Eight test tubes; test tube rack; piece of glass.

MATERIAL. Four strips of zinc, 10 × 1 cm.; four copper strips, 10 × 1 cm.; iron nails; solutions of lead acetate, copper sulphate, silver nitrate, mercury nitrate, and zinc nitrate.

(*a*) In separate test tubes place solutions of lead acetate, copper sulphate, silver nitrate, and mercury nitrate. In each tube place a strip of zinc, bending it over the lip of the tube (Figure 49). Let each tube stand for at least 5 minutes without disturbance.

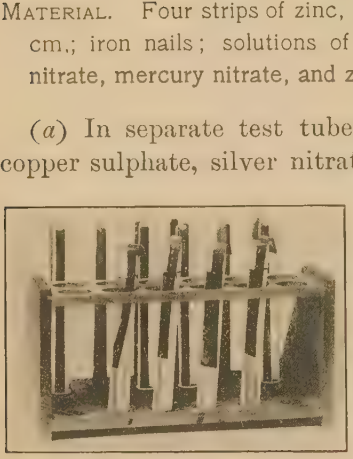


Figure 49.

(*b*) In a similar manner, place strips of copper in another set of test tubes, containing lead acetate, silver nitrate, zinc nitrate, and mercury nitrate.

(*c*) Remove the zinc strip that has been in the solution of mercury nitrate. Rinse the strip, and then rub it with the finger. Bend the strip sharply, and observe the broken edge.

What metal has been deposited on the zinc?

(*d*) Remove the copper strip from the solution of mercury nitrate. Rinse the strip, and rub it between the fingers.

What has been deposited on the copper?

What does the color of the solution remaining in the test tube indicate?

(*e*) Remove the zinc strip from the silver nitrate, scraping off the deposit on it into the test tube. Carefully pour off the liquid and fill the test tube with water. Shake the tube

and allow the deposit to settle. Pour off the water again. Gather the solid together, squeezing it into a pellet. Lay it on something hard and rub it with a piece of glass.

What metal was deposited on the zinc?

(*f*) To recognize very clearly the metal that is deposited from the copper sulphate solution, place an iron nail in a copper sulphate solution.

Result?

(*g*) Examine all the other strips and tabulate results.

TABLE

Metal Strip	Solution	Deposit

Write equations for all cases where a replacement actually occurs, but not for the others.

Explain why these reactions occur (*see Experiment 28*).

Assuming that one gram of silver was deposited, what would have been the weight of copper passing into the solution?

EXPERIMENT 65

Equivalent of Silver

APPARATUS. Beaker; balance; set of weights; wash bottle (Figure 50); glass rod with rubber tubing on the end; funnel; test tube; ring-stand with one ring.

MATERIAL. Silver nitrate solution, 34 grams per liter; piece of copper, 3 × 6 cm.; thread; filter paper; ammonium hydroxide, 1 to 3.

Record weighings in a table like that on page 180.

(*a*) Weigh accurately on a balance a piece of clean bright copper. Tie a piece of thread about it, curve it slightly, and suspend it in a beaker containing enough silver nitrate solution to completely cover it. Allow the copper to remain in the solution overnight.

Write the equation for the replacement that occurs.

(*b*) The next laboratory period, loosen the lightly adhering silver from the copper, and lift the strip from the solution by the thread. Every particle of the silver must be washed from the copper into the beaker. It may be necessary to rub the copper gently with the end of a glass rod covered with a bit of rubber tubing. The deposit does not adhere tightly and is more easily removed by gentle treatment than by hard rubbing.

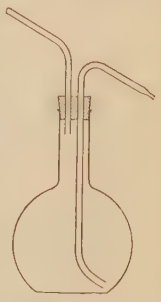


Figure 50.

A wash bottle (Figure 50), by which a fine jet of water can be directed against the copper, will be of great assistance in getting the silver into the beaker.

Wipe the copper strip, dry it at the ordinary temperature, and weigh it.

What does the change in weight represent?

(*c*) While the copper is drying, carefully filter the contents of the beaker on a *weighed* filter paper.[1] Be sure that every particle of the silver is transferred to the filter paper. The wash bottle will be of great help in this operation also.

The silver must now be thoroughly washed with water. Cover the deposit on the filter paper with water and allow it to drain completely before adding another portion. Wash in this way at least three times, or oftener, until a few drops of the filtrate falling into a little ammonia water in a test tube show no coloration.

Take at least twenty-four hours to dry the filter unless the heat of an air bath or a steam radiator is available, but the final drying must be done at the temperature of the laboratory.

When dry, weigh the filter paper and its contents.

TABLE

Original weight of piece of copper	g.
Final weight of piece of copper	g.
Weight of copper used	g.
Weight of filter paper with silver	g.
Weight of filter paper (obtained from instructor) . . .	g.
Weight of silver deposited	g.

Knowing the weight of the metals used, calculate the number of grams of silver that would be replaced by one gram of copper.

Taking 31.8 grams of copper as chemically equivalent to one gram of hydrogen, calculate the weight of silver that will be equivalent to one part by weight of hydrogen.

[1] The weight of the filter paper can be ascertained by weighing the entire pack, and then dividing the weight by the number of sheets.

CALCULATIONS

EXPERIMENT 66

Tests for Iron Salts

APPARATUS. Six test tubes; test tube rack.

MATERIAL. Solutions of ferrous sulphate or other soluble ferrous salt (freshly prepared and thoroughly reduced), ferric chloride, potassium ferrocyanide, potassium ferricyanide (freshly prepared).

These tests involve the use of two substances that are complex cyanides. Potassium ferrocyanide, $K_4Fe(CN)_6$, is a compound of potassium cyanide, KCN, and ferrous cyanide, $Fe(CN)_2$. Potassium ferricyanide, $K_3Fe(CN)_6$, is a compound of potassium cyanide, KCN, and ferric cyanide, $Fe(CN)_3$. Note that the valence of iron in each of the compounds can be found by subtracting the number of potassium atoms from the number of cyanogen groups, and that the name of each of the compounds indicates the valence of the iron it contains. These two compounds ionize according to the following equations:

$$K_4Fe(CN)_6 \rightleftarrows 4\,K^+ + Fe(CN)_6^{----}$$
$$K_3Fe(CN)_6 \rightleftarrows 3\,K^+ + Fe(CN)_6^{---}$$

In writing the formulas of compounds that contain these complex ions it is sometimes desirable to use brackets, as in the formula for ferric ferrocyanide: $Fe_4[Fe(CN)_6]_3$.

(*a*) To 5 cc. of a solution of ferrous sulphate (or other ferrous salt) add a few drops of potassium ferrocyanide solution.

Record the color of the precipitate in the table.

To 5 cc. of a solution of ferric chloride (or other ferric salt) add a few drops of potassium ferrocyanide.

Record the color of the precipitate in the table.

This substance is known as Prussian blue.

Write the equation for its formation.

To a mixture of solutions of ferrous sulphate and ferric chloride add a few drops of potassium ferrocyanide solution.

Which color obscures the other?

Is potassium ferrocyanide a test for the ferrous or for the ferric ion?

(*b*) To 5 cc. of a solution of ferrous sulphate (or other ferrous salt), add a few drops of potassium ferricyanide.

Record the color of the precipitate in the table.

This is known as Turnbull's blue.

Write the equation for its formation.

To 5 cc. of a solution of ferric chloride (or other ferric salt), add a few drops of potassium ferricyanide. Fill the test tube with water, shake the contents, and examine the mixture closely, to see whether the liquid is clear or a precipitate is formed.

Record the result in the table.

To a mixture of solutions of ferrous sulphate and ferric chloride, add a few drops of potassium ferricyanide.

Is the precipitate due to the ferrous or to the ferric salt?

Is potassium ferricyanide a test for the ferrous or for the ferric ion?

TABLE

Iron Salts	Potassium Ferrocyanide, $K_4Fe(CN)_6$	Potassium Ferricyanide, $K_3Fe(CN)_6$
Ferrous sulphate $FeSO_4$		
Ferric chloride $FeCl_3$		

EXPERIMENT 67

Action of a Reducing Agent on a Ferric Salt

APPARATUS. Three test tubes; bunsen burner; test tube holder; single-hole rubber stopper to fit test tube, provided with a short glass tube drawn to a capillary.

Figure 51.

MATERIAL. Solution of ferric chloride; dilute hydrochloric acid, 1 to 4; steel wool or fine iron wire; potassium ferrocyanide solution.

To 10 cc. of a solution of ferric chloride contained in a test tube, add a small bundle of steel wool or fine iron wire and 3 cc. of dilute hydrochloric acid. Close the tube with a rubber stopper containing a tube that ends in a capillary (Figure 51). Heat the tube gently.

From time to time pour a very small sample of the contents of the tube into a second test tube that contains a few drops of potassium ferrocyanide, using a fresh portion of ferrocyanide for each test.

Results?

What change is indicated by these tests?

Continue the boiling until the tests indicate that the change is complete.

Show by an equation what is produced by the reaction between the iron and the dilute hydrochloric acid.

Show by a second equation how one of these products reacts with the ferric chloride.

What change occurs in the valence of the iron ion?

The terms oxidation and reduction are, on this account, sometimes extended to mean change of valence.

When used in this way, does reduction imply a raising or a lowering of valence of the positive ion?

EXPERIMENT 68

Action of an Oxidizing Agent on a Ferrous Salt

APPARATUS. Three test tubes; bunsen burner.

MATERIAL. Solution of ferrous sulphate; dilute sulphuric acid, 1 to 6; hydrogen peroxide; concentrated nitric acid; solutions of potassium ferrocyanide and potassium ferricyanide.

To 5 cc. of a solution of ferrous sulphate, add 1 cc. of dilute sulphuric acid and 2 cc. of hydrogen peroxide. Test small samples of the resulting solution for ferrous ions and for ferric ions.

Results?

Complete the equation:

$$FeSO_4 + H_2SO_4 + H_2O_2 \longrightarrow _____ + _____ .$$

What change occurs in the valence of the iron ions?

Why is this change sometimes spoken of as oxidation?

Try the effect of nitric acid on a mixture of ferrous sulphate and dilute sulphuric acid. Boil the contents of the tube, and test for both ferrous and ferric ions.

Results?

Complete the equation:

$$FeSO_4 + H_2SO_4 + HNO_3 \longrightarrow _____ + _____ + NO$$

When used to indicate a change of this kind, does oxidation imply a raising or a lowering of the valence of the positive ion?

EXPERIMENT 69

Iron Salts in Photography — Blue Prints

APPARATUS. Two enameled pans, shallow, 8 × 10″; test tubes; graduate, 100 cc.

MATERIAL. Solution of ammonium ferric citrate (green scales), 100 g. in 1 liter; ferric chloride solution, 4 g. in 100 cc.; potassium ferricyanide solution, 100 g. in 1 liter; oxalic acid solution, 50 g. in 1 liter; paper, unglazed, of good quality, cut into sheets 3″ × 4″.

(*a*) Prepare two pieces of sensitive paper. This should be done in a darkened room, closet, or cupboard. Holding the paper by a corner, draw it lightly across the surface of a solution of ammonium ferric citrate contained in a shallow pan (Figure 52). Avoid getting the back of the paper wet with the solution, and make sure that the face is completely and evenly moistened. Hang the paper to dry in a dark place.

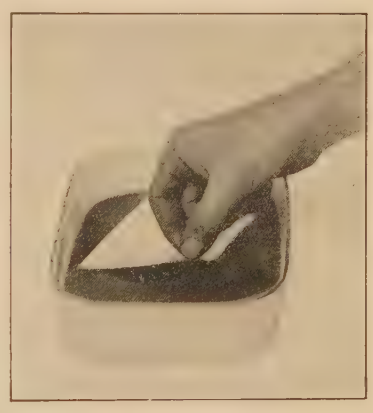


Figure 52.

(*b*) In a darkened room or cupboard, mix in a test tube 3 cc. of ferric chloride with 3 cc. of oxalic acid solution. Divide this mixture into two parts. Keep one part in the dark, while exposing the other to the direct rays of the sun for several minutes. To each of the tubes then add 3 cc. of a solution of potassium ferricyanide.

In which tube has a ferrous salt been produced?

Complete the equation:

$$FeCl_3 + H_2C_2O_4 \longrightarrow _____ + \quad HCl + \quad CO_2$$

This is an example of a chemical action produced by light.

Which of the substances may be regarded as having been reduced?

Which may be regarded as the reducing agent?

(*c*) Ammonium ferric citrate makes an excellent substance for preparing blue print paper, because it contains within itself both the iron salt and the reducing agent.

When the paper prepared with ammonium ferric citrate at the beginning of the period is thoroughly dry, arrange it for exposure to sunlight so that an opaque object, such as a key, or a stencil cut in heavy paper, will keep the light from striking part of the paper. The exposure should last three to ten minutes according to the strength of the light. At the end of this time, examine the paper quickly, and then immerse it, face down, in a pan containing a solution of potassium ferricyanide.

Was any change apparent before the paper was put into the solution?

After it was put into the solution?

Complete the equation:

$$FeCl_2 + K_3Fe(CN)_6 \longrightarrow _____ + _____$$

Wash the paper thoroughly in running water, dry it, and paste it in the place provided on the next page.

What purpose does potassium ferricyanide serve in this operation?

What common photographic term might be applied to the potassium ferricyanide?

Why is it necessary to wash the paper thoroughly?

What photographic term can be applied to this part of the operation?

SAMPLE PRINT

EXPERIMENT 70

Silver Salts in Photography

APPARATUS. Three test tubes, which must be clean and entirely free from grease; small graduate.

MATERIAL. Solutions of silver nitrate (17 grams per liter), potassium bromide (36 grams per liter), hypo (25 per cent solution); developer as follows: 1600 cc. water, 10 grams hydrochinone, 20 grams sodium sulphite (dry); 1 gram potassium bromide, 1 gram citric acid, 20 grams sodium carbonate (dry); or the ordinary developing powders may be used, diluting according to directions for tank use; dark paper.

Protect the materials from the light, by wrapping a dark paper about the test tubes. Do not warm with the hands. All materials must be pure and the test tubes *must be clean.*

In each part, (*a*), (*b*), and (*c*), suspended silver bromide is prepared by adding to a test tube one fourth full of water, *not more than* 1 cc. of potassium bromide solution and an exactly equal amount (1 cc.) of silver nitrate solution; mix *well*, but do *not* shake hard. (If shaken hard, the bromide will become lumpy.)

(*a*) Expose silver bromide to the light for a few minutes, shaking it to expose all parts equally. (The bromide may change color, if impure.)

To the exposed bromide, add about 5 cc. of the developing solution. Allow the action to continue for two minutes.

Result?

Then add about 10 cc. of hypo solution (fixer), and shake well.

Result?

(*b*) To the suspended silver bromide (well protected from the light) add 5 cc. of developer. Keep in the dark for two minutes.

Result?

Then add about 10 cc. of the fixer.

Result?

(*c*) To the suspended silver bromide add about 10 cc. of the fixer.

Result?

TABLE

	Silver Bromide Exposed to Light	Silver Bromide not Exposed to Light
Action of developer		
Action of fixer		

The *developer* is a reducing agent capable of *continuing, but not initiating*, the reduction of a silver salt.

Under what conditions is an insoluble residue (silver) obtained?

"Hypo" is the last solution used in the preparation of a negative.

What is its action?

EXPERIMENT 71

Aluminum Hydroxide

APPARATUS. Hydrometer jar, or cylindrical graduate, 250 cc.; three test tubes; glass stirring rod, 15″.

MATERIAL. Aluminum sulphate, 20 g. to the liter; limewater; fine clay; ammonium hydroxide solution, 1 to 10; logwood solution; alizarine mixed with water.

(*a*) **Preparation.**

To one sixth of a test tube of a dilute solution of aluminum sulphate, $Al_2(SO_4)_3$, add twice as great a volume of limewater.

Result?

What is the appearance of this precipitate of aluminum hydroxide?

Write the equation for the precipitation.

(*b*) **As a coagulum.**

Add water to a cylindrical jar until it is about two thirds full. Render the water turbid by stirring in a little fine clay. Then pour in one third of a test tube of aluminum sulphate solution and mix it with the turbid water by thorough stirring.

To this mixture, add slowly, without stirring, two thirds of a test tube of limewater. Allow the water to stand.

Note what is happening from time to time.

Result?

What precipitate was formed in the turbid water?

How are the suspended particles of clay removed so as to leave the water clear?

(*c*) **Formation of lakes.**

To one sixth of a test tube of logwood solution, add ammonium hydroxide. Note that no precipitate is formed, although the logwood changes somewhat in color.

To a solution of aluminum sulphate in another test tube, add some ammonium hydroxide.

What is the precipitate?

Add some logwood solution to the test tube containing the precipitate. Shake the test tube and allow the contents to settle.

What does the color of the liquid in the test tube show about the amount of logwood in solution?

Compare the color of the precipitate with the color of logwood and with the color of aluminum hydroxide.

Such a combination of a dye with aluminum hydroxide or other suitable compound, is commonly called a "lake."

Formerly aluminum hydroxide was extensively used in dyeing cloth. Its gelatinous nature enables it to adhere to the fibers of the cloth. It also adheres to the dye. Thus it acts as a binder between the cloth and the dye. A substance that has this characteristic is said to be a *mordant*.

State how you would dye a piece of white cotton cloth by using the same three solutions employed to make the logwood lake.

Using aluminum sulphate solution, ammonium hydroxide solution, and alizarine, make an alizarine lake.

How does the alizarine lake differ in color from the original alizarine and from the aluminum hydroxide?

EXPERIMENT 72

Substantive, Salt, or Direct Colors for Cotton

APPARATUS. Five agateware cups or stewpans, 1 pt.; two ring-stands with ring; two bunsen burners; balance sensitive to 0.1 g. and weights; yard-stick; shears; funnel, $3\frac{1}{2}''$; three stirring rods; two test tubes, 6"; graduate, 100 cc.

MATERIAL. Sodium carbonate solution, 2 g. Na_2CO_3 per liter; white cotton cloth (cheesecloth); white woolen yarn; saturated solution of plaster of Paris; saturated solution of salt; stock solution of Congo red, 5 g. to the liter; stock solution of sodium chloride, 50 g. to the liter; Castile soap; filter paper.

(*a*) Weigh a piece of unbleached cotton cloth (cheesecloth) and also determine the number of square inches it contains. Calculate the number of square inches weighing approximately 10 g. and cut the cloth into strips containing this number of square inches.

Figure 53.

(*b*) To aid in obtaining an even color, the material to be dyed should be clean and saturated with water before being placed in the dye bath. The treatment preparatory to dyeing varies with the kind of goods, the impurities adhering to the fiber, the shade to be obtained, and the nature of the dye.

Pour into a pint agateware cup (Figure 53) 200 to 300 cc. of the solution of sodium carbonate, add two strips of cloth, and bring the liquid to a gentle boil. Continue to boil the cloth gently until it is required for use in part (*f*).

Take a skein of white woolen yarn weighing approximately

5 g. and boil it in suds made by dissolving Castile soap in warm water. Let gentle boiling continue until the yarn is required for use in part (*f*).

(*c*) Pour 5 cc. of a saturated solution of plaster of Paris into a test tube and add a few drops of a 0.5% solution of Congo red. Mix the liquids thoroughly and then filter.

Describe the substance left on the filter.

Congo red is the sodium salt of an acid. The calcium and magnesium salts of this acid are insoluble and, therefore, are formed when Congo red is added to the hard water. A similar action takes place with most of the substantive colors.

Why should a hard water be softened before being used in a dye bath?

(*d*) To 5 cc. of a nearly saturated solution of salt in a test tube, add a few drops of a 0.5% solution of Congo red. Thoroughly mix the contents of the tube and allow it to stand for a few minutes, then filter.

What do you observe concerning the color of the filtrate?

The Congo red, being less soluble in the salt solution than in pure water, was precipitated. This process is technically termed "salting out."

(*e*) In preparing a dye bath, the quantity of dye used depends upon the weight of the material to be colored, the color used, and the shade to be obtained. In three agateware vessels prepare the following baths:

Bath # 1. Dissolve in 300 cc. of soft water a quantity of Congo red equal in weight to 2 per cent of the weight of one of the pieces of cotton cloth. This weight is calculated as follows:

1 cc. of the stock solution of Congo red contains 0.005 g. of Congo red. Each piece of cloth weighs 10 g. Two per cent of 10 g. is 0.2 g. Your bath should contain 0.2 g. of Congo red, or $\frac{0.2}{0.005} = 40$ cc. of the stock solution.

Bath # 2. To 300 cc. of water, add 40 cc. of the stock solution of Congo red and sodium chloride equal in weight to 20 per cent of a piece of cloth. 1 cc. of the stock solution of salt contains 0.05 g. of sodium chloride.

How many cubic centimeters of the stock solution of sodium chloride should be used?

Bath # 3. To 300 cc. of water, add Congo red equal in weight to 2 % of a skein of wool (5 g.) and salt equal to 20 % of the weight of the skein.

Calculate and record the volume of the stock solutions required.

(*f*) Remove the cotton cloth and the woolen yarn from the baths used in part (*b*). Thoroughly rinse the cotton and the wool in soft water and then distribute the material as follows:

Place a strip of cotton cloth in Bath # 1.

Place a strip of cotton cloth in Bath # 2.

Place a skein of woolen yarn in Bath # 3.

Heat the baths to boiling and continue gentle boiling for fifteen minutes. During the boiling, frequently move the goods in the baths so as to make sure that the dye comes in contact with every part of the material.

(*g*) Remove the goods from the baths, wash thoroughly in water, and then dry. Examine the liquid remaining in the agateware vessels.

Did the cotton or the wool extract the more color from the bath?

Compare the color of the cloth dyed in the bath containing no salt with that dyed in the bath containing salt.

In which case was the more dye deposited on the goods?

After referring to part (d) account for this fact.

(*h*) Place a piece of white cloth and a piece of colored cotton cloth in a dish half filled with water and boil for a few minutes.

Result?

Colors that behave in this way are said to "bleed"; that is, they are not fast in washing.

Direct colors vary greatly in their fastness to washing, their fastness to light, and their comparative affinity for cotton and wool.

Attach each piece of cloth as an exhibit in the place provided for it.

SAMPLES DYED IN CONGO RED

Cotton, Bath No. 1	Cotton, Bath No. 2	Wool, Bath No. 3	Samples, Part (*h*)

EXPERIMENT 73

Acid Colors

APPARATUS. Two agateware vessels, 1 pt.; ring-stand with ring; bunsen burner; two glass stirring rods; thermometer; pipette, 10 cc.; balance with weights; graduate, 100 cc.

MATERIAL. Two pieces of cotton cloth (cheesecloth) weighing 5 g. each; two skeins white woolen yarn weighing 5 g. each; stock solution of Biebrich scarlet, tartrazine, or acid blue containing 5 g. to the liter (dissolve the color in hot water); stock solution of Glauber's salt (cryst. sodium sulphate) containing 50 g. to the liter; stock solution of sulphuric acid made by dissolving 55 cc. of sulphuric acid (sp. gr. 1.84) in water and diluting to 1 liter; picric acid; sodium carbonate; Castile soap.

(*a*) Prepare 5 g. samples of cotton cloth and of woolen yarn for the dye bath by boiling the cotton in a dilute solution of sodium carbonate and the wool in a soap solution as directed in Experiment 72.

(*b*) Dissolve 0.1 g. of picric acid in 300 cc. of water in an agateware vessel. Warm to 60° C. and then add a sample of cotton cloth and one of woolen yarn. Raise the temperature of the bath to near its boiling point. Remove the samples and wash them thoroughly.

Results?

(*c*) The acid colors are nearly all salts of color acids from which the color acid is liberated, during the process of dyeing, by the addition of another acid, generally sulphuric, to the bath. More sulphuric acid than the amount necessary to liberate the color acid is added to the bath, because the excess of sulphuric acid acts on the wool and causes it to have a greater tendency to combine with the dye. The addition of sodium sulphate to the bath generally improves the evenness of

the deposition of color on the fiber; its chief action probably is to retard the rate of liberation of the color acid.

Prepare a dye bath by adding to 300 cc. of water, 2 cc. of the stock solution of sulphuric acid, 10 cc. of the stock solution of Glauber's salt, and 10 cc. of the stock solution of color.

How many grams does the bath contain (a) of sulphuric acid? (b) of Glauber's salt? (c) of color?

The bath contains the usual quantities used for dyeing 5 g. of wool.

What per cent of the weight of the wool is (a) the sulphuric acid? (b) the Glauber's salt? (c) the color that is used?

Warm the bath to 60° C., and add 5 g. of woolen yarn. Raise the temperature of the bath to near its boiling point and continue that temperature for ten minutes, meanwhile working the goods in the bath. Remove the wool from the bath and wash it thoroughly. Into another bath, prepared in a similar manner, put 5 g. of cotton cloth and heat it as you did the woolen yarn.

To which kind of material, cotton or wool, does the dye adhere more firmly?

Acid colors are used chiefly in dyeing woolen and silk goods. As a class, they are fast to light, but not to washing, and consequently are suitable for dyeing goods not intended to be washed. A few of them are fast to washing, and some of them dye wool but not silk.

Attach a sample of each of the pieces of cloth in the place provided for it.

SAMPLES

Cotton, Picric Acid	Wool, Picric Acid	Cotton, Part (*c*)	Wool, Part (*c*)

EXPERIMENT 74

Basic Colors

APPARATUS. Two beakers, 200 cc.; two agateware vessels, 1 pt.; ring-stand with ring; bunsen burner; three glass stirring rods; 100 cc. graduate.

MATERIAL. Cheesecloth, 10 g. pieces; blue litmus paper; standard tannic acid solution, 10 g. per liter; standard solution of tartar emetic, 10 g. tartar emetic per liter; standard solution of malachite green, 5 g. malachite green per liter; standard solution of Fuchsine, 5 g. Fuchsine per liter; acetic acid; soft or distilled water.

Basic colors are direct colors for wool and silk, but do not adhere to cotton. Cotton, however, can be treated with substances, called mordants, that cling to the fiber and also to the color. By the use of mordants the color is held to the cotton fiber.

(*a*) Add 20 cc. of the standard solution of tannic acid to 100 cc. of water in a 200 cc. beaker and heat nearly to boiling. If the water used is hard, add, previous to the dissolving of the tannic acid, sufficient acetic acid to give a slightly acid reaction with litmus.

Put 10 g. of cheesecloth in the hot solution of tannic acid and allow it to remain for at least two hours, allowing the solution of tannic acid to cool meanwhile. If more convenient, allow the cheesecloth to remain in the tannic acid bath until the next laboratory period.

(*b*) Pour 100 cc. of water into a beaker and add 10 cc. of the standard solution of tartar emetic. Remove the cloth that has been in the tannic acid solution a sufficient length of time, squeeze it, and put it into the cold tartar emetic bath.

The tartar emetic combines with the tannic acid to form an insoluble salt that adheres to the cotton fiber.

(*c*) Prepare in the agateware vessels two duplicate dye baths. In each case, add to 200 cc. of water sufficient acetic

acid to make the bath slightly acid, and then add 40 cc. of the standard solution of malachite green.

The acetic acid serves the double purpose of correcting any hardness that may be in the water, and of preventing a too rapid dyeing of the goods that might result in unevenness of shade.

In one dye bath, place 10 g. of cheesecloth that has not been mordanted.

Take a piece of cheesecloth from the tartar emetic and wash it thoroughly in soft water. Then put the cheesecloth into the dye bath not in use.

Slowly heat the two dye baths to about 60° C. and dye the pieces of cloth for ten minutes or more, meanwhile moving them in the bath so as to expose all parts to the action of the dye.

Remove the pieces of cloth from the dye baths and wash them thoroughly with water, being careful to use different portions of water for each piece of cloth.

To which piece of cloth does the dye adhere the more firmly?

Divide into two equal parts the piece of cloth that has been mordanted and then dyed. Dip one of them in a dye bath made by adding 4 cc. of a standard solution of Fuchsine to 300 cc. of water that was slightly acidified by acetic acid. Remove the cloth as soon as it becomes thoroughly wet by the Fuchsine solution, wash, and then note the effect of the red on the brilliancy of the green.

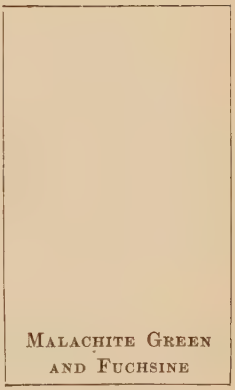
Result?

Attach a sample of each of the pieces of cloth in the space provided for it.

SAMPLES

UNMORDANTED MALACHITE GREEN

MORDANTED MALACHITE GREEN



MALACHITE GREEN AND FUCHSINE

EXPERIMENT 75

Double Salts

APPARATUS. 100 cc. flask; 250 cc. beaker; two beakers, 100 cc.; stirring rod; balance and weights; funnel; tripod; bunsen burner; wire gauge with asbestos center; graduate.

MATERIAL. The material for the preparation of each salt will be found accompanying the directions for making the salt.

(*a*) **Preparation of ferrous ammonium sulphate,**

$$FeSO_4(NH_4)_2SO_4 \cdot 6\,H_2O.$$

MATERIAL. Filter paper; iron wire or nails; ammonium hydroxide; concentrated sulphuric acid; sulphuric acid, 1 : 10; red and blue litmus papers.

Take 50 cc. of dilute sulphuric acid in a flask and dissolve in it clean iron wire as long as hydrogen is given off.

Write the equation for the formation of ferrous sulphate, $FeSO_4$.

While waiting for the wire to dissolve, take another 50 cc. of dilute sulphuric acid in a beaker and neutralize with ammonium hydroxide solution.

What salt is formed?

Write the equation.

Filter the ferrous sulphate solution into the solution of ammonium sulphate.

Evaporate the mixed solution to one third its volume. Add a few drops of the concentrated sulphuric acid and set the solution aside to crystallize.

Pour off the liquid into the bottle designated by the instructor. Empty the crystals of the ferrous ammonium sulphate on a filter paper in a funnel. When the crystals have

drained, dry them between sheets of filter paper. They can be used for making the test for nitrates.

Write the equation for the formation of ferrous ammonium sulphate.

(*b*) **Preparation of potassium alum,** $KAl(SO_4)_2 \cdot 12\,H_2O$.

MATERIAL. Filter paper; potassium sulphate; aluminum sulphate.

Write the equation for the preparation of potassium alum from potassium sulphate and aluminum sulphate.

Weigh out 15 grams of crystallized aluminum sulphate,

$$Al_2(SO_4)_3 \cdot 18\,H_2O.$$

Calculate from the equation how many grams of potassium sulphate are needed.

Weigh out the calculated amount of potassium sulphate and dissolve in 40 cc. of hot water.

In another beaker dissolve the aluminum sulphate in 40 cc. of hot water.

Mix the two solutions and set the mixture aside to crystallize in a place where the beaker will not be disturbed.

(*c*) Ammonium alum, $(NH_4)Al(SO_4)_2 \cdot 12\,H_2O$.

MATERIAL. Crystallized aluminum sulphate; ammonium hydr
sp. gr. 0.90; sulphuric acid, 1 : 10; filter paper; red and blue l
papers.

Weigh out 15 grams of aluminum sulphate, and dissolve
40 cc. of water.

Add 4.5 cc. of ammonium hydroxide solution to 15 c
water, neutralize it with dilute sulphuric acid, and then d
it to about 40 cc.

Mix the two solutions, filter the mixture if it is not clear
evaporate the solution to half the bulk.

Set the solution aside to crystallize.

Describe the color and general form of the crystals.

Write the equations for the reactions taking place.

(*d*) Ferric ammonium alum, $(NH_4)Fe(SO_4)_2 \cdot 12\,H_2O$.

MATERIAL. Filter paper; ammonium hydroxide, iron wire or
sulphuric acid, 1 : 10; concentrated nitric acid; red and blue l
papers.

Prepare a solution of ferrous sulphate as in part (*a*).

Prepare a solution of ammonium sulphate as in part (*a*)

To the filtered solution of ferrous sulphate in a beaker
25 cc. of the sulphuric acid, and 1 cc. of concentrated nitric
Boil as long as nitric oxide is evolved.

Into what gas is nitric oxide converted on coming in contact wit

The iron is now in solution as ferric sulphate, $Fe_2(SO_4)$

Write the equation for the reaction.

Add the solution of ammonium sulphate to the solution of ferric sulphate, evaporate until the bulk is reduced to one half. Set the beaker aside to cool slowly until the contents crystallize.

Describe the color and general form of the crystals.

The crystals may be dried between blotters; they must be preserved in well-stoppered bottles.

Write the equation for the reaction between the sulphate of iron and the ammonium sulphate.

(*e*) **Rochelle salt,** $NaK(C_4H_4O_6) \cdot 4\,H_2O$.

MATERIAL. Cream of tartar; bicarbonate of soda; filter paper.

Weigh out 14 g. of acid potassium tartrate (cream of tartar), $KH(C_4H_4O_6)$. Mix it with 15 cc. of water.

Does it completely dissolve in this quantity of water?

Weigh out 6 g. of sodium bicarbonate, $NaHCO_3$, and mix it with 15 cc. of water.

Does it completely dissolve in this quantity of water?

While stirring, slowly pour the sodium bicarbonate solution into that of the tartrate, and pour back from beaker to beaker until action ceases.

What gas is evolved?

Is all the solid dissolved?

If the liquid is not clear, filter it, and evaporate it to half bulk and set it aside to crystallize. Better crystals may be obtained by setting it aside to evaporate without heating, but do not let it evaporate to dryness. Pour off the mother liquor and dry the crystals between blotters or filter paper.

Write the equation for the reaction between sodium bicarbonate and cream of tartar.

(*f*) **Rochelle salt**, $NaK(C_4H_4O_6) \cdot 4\ H_2O$.

MATERIAL. Tartaric acid; sodium hydroxide, 1 : 10; potassium hydroxide, 1 : 10; phenolphthalein paper.

Dissolve 5 g. of tartaric acid in 10 cc. of water. Exactly neutralize it with sodium hydroxide.

Dissolve 5 g. of tartaric acid in 10 cc. of water. Exactly neutralize it with potassium hydroxide.

Mix the two solutions and evaporate to one half the bulk or, to get good crystals, set it aside to evaporate slowly to one fourth the bulk. Pour off the mother liquor and dry the crystals between blotters or filter paper.

Write the equation for the reaction between sodium hydroxide and tartaric acid, $H_2(C_4H_4O_6)$.

Write the equation for the reaction between potassium hydroxide and tartaric acid.

Write the equation for the reaction which resulted in the formation of the Rochelle salt.

(*g*) **Ammonium sodium tartrate.**

MATERIAL. Tartaric acid; ammonium hydroxide; sodium hydroxide; phenolphthalein paper.

Proceed as above, but use ammonium hydroxide instead of potassium hydroxide. Ammonium sodium tartrate does not crystallize as well as Rochelle salt.

Write equation as in part (*f*).

EXPERIMENT 76

Qualitative Separation of Lead, Silver, and Mercury

APPARATUS. Test tubes; test tube rack; funnel; bunsen burner.

MATERIAL. Hydrochloric acid concentrated and dilute; nitric acid concentrated and dilute; ammonium hydroxide 1 to 3; solutions of lead, silver, and mercurous nitrates; potassium chromate solution; copper strip; filter paper; unknown solutions.

(*a*) In one test tube take 10 cc. of a solution of lead nitrate; and in another, 10 cc. of a solution of silver nitrate. To both test tubes add dilute hydrochloric acid till the reaction is complete.

Results?

Write the equations.

Allow the precipitates to settle and then pour off the supernatant liquid from each of the two test tubes. Add to the precipitates in the test tubes enough cold water to nearly fill the tubes, and shake the contents. Again let the precipitates settle and then pour off the supernatant liquids.

What compound has been removed by washing the precipitate and then pouring off the supernatant liquid?

Try the effect of hot water on the precipitate of lead chloride.

Result?

Divide the precipitate of silver chloride between two test tubes. With one part try the effect of hot water; with the other, the effect of ammonium hydroxide.

Results?

(*b*) To 10 cc. of a solution of mercurous nitrate, $HgNO_3$, add dilute hydrochloric acid till the action is complete.

Result?

Write the equation.

Wash the precipitate with cold water and divide it between two test tubes. Find out whether hot water dissolves the mercurous chloride.

Result?

What effect does ammonium hydroxide have upon the mercurous chloride?

(*c*) In the same test tube take 5 cc. each of solutions of silver, lead, and mercurous nitrates. Add dilute hydrochloric acid till precipitation is complete.

Of what does the precipitate consist?

Filter. Wash the precipitate on the filter paper with a very little *cold* water. Next wash the precipitate thoroughly with hot water, keeping the washings.

Which one of the chlorides was dissolved by the hot water?

To confirm this, add to the hot filtrate a solution of potassium chromate, K_2CrO_4. This chromate gives an insoluble and characteristic compound of the metal whose chloride is soluble in hot water.

Write the equation for this confirmatory test.

Give name, formula, and color of the characteristic compound formed.

Wash the precipitate remaining on the filter paper with ammonium hydroxide, keeping the washings.

Which chloride gives the color?

What chloride is contained in the ammonium hydroxide filtrate?

Prove the presence of this chloride by adding a slight excess of nitric acid.

Name the precipitate and state the characteristic properties by which you recognize it.

To dissolve the precipitate still remaining on the filter paper, add a little *aqua regia* (9 drops concentrated hydrochloric acid to 3 drops concentrated nitric acid). Dilute with water the solution thus obtained, and put into it a bright strip of copper. After several minutes, remove the strip, and wash and rub it.

Result?

Explain why this dissolving in aqua regia and the addition of a copper strip is a confirmatory test.

(*d*) Obtain from the instructor an unknown solution. Using the methods in (*c*), analyze the solution for lead, silver, and mercury.

Record all the steps, even those giving negative results.

Underline the metal found in your unknown.

lead mercury silver

EXPERIMENT 77

Chromium Compounds

APPARATUS. Two beakers, 250 cc.; two test tubes; graduate; balance and weights; bunsen burner.

MATERIAL. Potassium dichromate; potassium hydroxide; sodium peroxide; chromium sulphate or chrome alum; alcohol; dilute nitric acid; concentrated sulphuric acid.

(*a*) Pulverize 10 grams of potassium dichromate and dissolve it in 50 cc. of water. The color of the solution is characteristic of dichromate ions.

(*b*) Dissolve 10 grams of potassium hydroxide in 100 cc. of water.

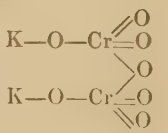
(*c*) Slowly add, with constant stirring, the solution prepared in (*b*) to that prepared in (*a*) until the resulting liquid is of a pure yellow color. The color is characteristic of chromate ions.

Complete the equation:

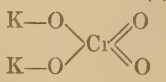
$$K_2Cr_2O_7 + 2\ KOH \longrightarrow 2\ K_2CrO_4 + _____$$

Any strong base would produce this change.

The structural formula for potassium dichromate is



The structural formula for potassium chromate is



What is the valence of chromium in each case?

(*d*) Add a dilute solution of nitric acid to the solution obtained in (*c*) until the color of the liquid shows that the chromate ions have been changed to dichromate ions.

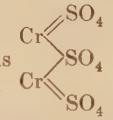
Write the chemical equation representing the change.

Why might a solution of any strong acid be used in place of the nitric acid?

(*e*) Drop by drop, pour 1 cc. of concentrated sulphuric acid into 5 cc. of water.

Dissolve 1 gram of powdered potassium dichromate in 10 cc. of water and then add 1 cc. of alcohol.

Pour the first solution into the second. Warm the mixture gently. The green color is due to chromic ions, from the compound chromic sulphate.

The formula for chromic sulphate is 

When a dichromate is changed into a chromic salt, is the change one of oxidation or of reduction?

Why?

(*f*) Add sodium peroxide, a little at a time, to 1 gram of chromium sulphate or chrome alum dissolved in 25 cc. of water, until the yellow color characteristic of chromate ions has been produced.

What element is liberated when sodium peroxide is added to water?

What base is formed?

What change takes place when a chromic salt is oxidized in the presence of a base?

EXPERIMENT 78

Fermentation

APPARATUS. Acid bottle, capacity about $2\frac{1}{2}$ liters; two wide-mouth bottles, capacity about 500 cc.; stoppers and tubes shown in Figure 54; test tube; boiling flask, 250 cc.; U-tube, 6″; battery jar, 5″ high; stoppers and tubes shown in Figure 55; thermometer; watch glass; beaker, 100 cc.; ring-stand with 1 ring and a small clamp; bunsen burner; wire gauze with asbestos center.

MATERIAL. Molasses; two yeast cakes; limewater; iodine; quicklime; sodium hydroxide solution; candle or splinter.

(*a*) **Fermentation.**

Arrange apparatus as shown in Figure 54. One set of apparatus will furnish sufficient fermented liquid for ten pupils to use in part (*b*). Pour 1 volume of molasses dissolved in 6 volumes of water into the acid bottle. Break two yeast cakes into small fragments and stir them into a little lukewarm water, so as to form a thin paste. Pour the paste into the bottle containing the solution of molasses, then shake the bottle so as to distribute the yeast through the molasses.

Figure 54.

After replacing the stopper in the acid bottle, remove the stopper from the wide-mouth bottle connected with the acid bottle, and fill the bottle with water. Replace the stopper. Some of the water will be forced into the third bottle, but no siphoning can take place since air cannot enter the acid bottle.

Allow the apparatus and contents to remain in a warm place for several days (about a week). From time to time, examine the contents of the bottles.

What change do you notice in the color of the liquid in the acid bottle?

What has happened to the water in the wide-mouth bottles?

While the action is in progress, remove the third bottle for a moment and replace it with a test tube containing a little lime-water so arranged that the end of the delivery tube will be in the limewater.

Account for the result.

After the action has been going on for several days, remove the stopper from the bottle filled with gas and lower into it a lighted candle, or burning splinter.

Results?

What is the name of the gas?

(*b*) **Fractional distillation.**

Arrange apparatus as shown in Figure 55.

Siphon off enough of the fermented liquid to half fill a distilling flask and clamp the flask in place. Insert the thermometer so that its bulb is in the neck of the flask just below the delivery tube.

Figure 55.

After connecting the condensing tube to the flask, bring the liquid in the flask to a gentle boil, then regulate the temperature very carefully, in order to prevent froth from being formed and passing over into the distillation tube.

Why should the water in the battery jar be changed occasionally or lumps of ice be put in the water?

At what temperature does the liquid commence to distil over?

When the temperature has reached 85°, take away the flame. Remove the U-tube from the water, wipe it dry on the outside, and pour enough of its contents into a watch glass to make a circle as big as a quarter. Hold a bunsen flame to the watch glass for a moment.

What happens?

What is shown to be present by this test?

After the action is over, is any liquid left in the watch glass?

Stop the distillation at 99° C.

At the close of the distillation try to ignite a few drops of the distillate.

Result?

Throw away the liquid in the flask.

Place in the distilling flask a number of small lumps of quicklime. Pour the first distillate back into the flask and redistil it, noticing the temperature of the vapor.

Why is the lime used?

Identify the second distillate (*a*) by its odor, (*b*) by bringing a lighted match to a small portion in a watch glass, and (*c*) by applying the iodoform test to another portion. To make the

iodoform test, add to the liquid to be tested a little sodium hydroxide solution, and then iodine, a crystal at a time, and warm. Stop the addition of the iodine before a permanent brown color is obtained. The iodoform will separate as a yellow precipitate and can be recognized by its odor.

During the fermentation, the cane sugar in the molasses was converted into a mixture of two simpler sugars, dextrose and levulose:

$$\underset{\text{cane sugar}}{C_{12}H_{22}O_{11}} + \underset{\text{water}}{H_2O} \longrightarrow \underset{\text{dextrose}}{C_6H_{12}O_6} + \underset{\text{levulose}}{C_6H_{12}O_6}$$

This change was brought about by a ferment (invertase) produced during the growth of the yeast plant. Then another ferment (zymase), also from the yeast, caused the change of the simpler sugars into ethyl alcohol, C_2H_5OH, and the gaseous product you tested.

Complete the equation for this change:

$$C_6H_{12}O_6 \longrightarrow _____ + _____$$

Ethyl alcohol boils at 78° C.

How do you explain the rise in the boiling point during the distillation?

EXPERIMENT 79

Preparation of Ethereal Salts (Esters)

APPARATUS. Bunsen burner; three test tubes; notched cork to fit test tube.

MATERIAL. Sulphuric acid, concentrated; sodium acetate; salicylic acid; ethyl alcohol, 95 per cent; amyl alcohol; methyl alcohol.

(*a*) Ethyl acetate.

Dissolve enough sodium acetate to fill the curved bottom of a test tube in a very little water, and then add a few drops of concentrated sulphuric acid. Acetic acid is produced, $H(C_2H_3O_2)$.

Complete the equation:

$$NaC_2H_3O_2 + H_2SO_4 \rightarrow Na_2SO_4 + _____$$

To the contents of the test tube add a few drops of ethyl alcohol. Warm the tube gently, and note a distinctive odor, different from that of alcohol. This is due to ethyl acetate, $C_2H_5 \cdot C_2H_3O_2$.

Write the equation for its formation.

(*b*) Amyl acetate.

Put 1 drop of amyl alcohol, $C_5H_{11}OH$, into a test tube, add two drops of concentrated sulphuric acid, and then add a small pinch of sodium acetate. Warm the test tube gently. Remove the tube from the flame, and, from a test tube with a notched cork, let water run, a drop at a time, down the side of the tube, until about 3 cc. have been added. Again warm the test tube, shaking it as you do so. Note the distinctive odor. Amyl acetate has been formed.

In what paint have you noted this odor?

Of what fruit does it remind you?

(*c*) **Methyl salicylate.**

Put a small pinch of salicylic acid into a test tube. Add a drop of methyl alcohol, and about 20 drops of concentrated sulphuric acid. Warm the mixture *very gently*, shaking the tube as you do so. Note the odor produced.

Of what familiar substance does it remind you?

(*d*) **General questions.**

What are the products of the reaction between a base and an acid?

What radical is common to bases and alcohols?

Show the similarity between an ester and an inorganic salt.

The formation of esters is known as *esterification*. This is easily reversed. The reverse action is known as *saponification*.

What must take place in order that a reaction may go to an end?

How does the presence of concentrated sulphuric acid aid in the formation of esters?

EXPERIMENT 80

Soap Making

APPARATUS. Bunsen burner; ring-stand; teaspoon; evaporating dish; graduate, 25 cc.; stirring rod.

MATERIAL. Beef suet (rendered by heating it in an evaporating dish until the fat is melted, and then pouring the fat off the connective tissue), or lard; 33 per cent solution of sodium hydroxide.

Place in an evaporating dish 2 level teaspoonfuls of "rendered" beef fat, or lard. Heat the dish with a small flame until the fat is melted. Then add 3 cc. of a 33 per cent solution of sodium hydroxide. Warm the dish very gently with a small flame, stirring the contents constantly. Continue the heating until there is a soapy mass in the dish. Then allow the dish to cool. The residue left in the dish is soap.

Beef fat is mainly glyceryl stearate, $C_3H_5(C_{18}H_{35}O_2)_3$. Stearic acid has the formula $H(C_{18}H_{35}O_2)$.

Write the equation for the reaction between the glyceryl stearate and the sodium hydroxide, producing sodium stearate (a soap) and glycerine, $C_3H_5(OH)_3$.

Organic fats and oils are esters of the alcohol glycerine and fatty acids.

How are such fats and oils converted into soap?

What alcohol is a by-product of soap making?

What became of it in the case of the soap just made?

EXPERIMENT 81

Starch

APPARATUS. Beaker, 150 cc.; three test tubes; glass stirring rod; funnel; porcelain evaporating dish; sand bath; ring-stand with large and small rings; wire gauze with asbestos center; bunsen burner.

MATERIAL. Starch; potassium iodide solution of iodine; glucose; Fehling solution; hydrochloric acid.

(*a*) Starch heated with water.

To one sixth of a test tube of water add a pinch of starch and shake the tube.

Does the uncooked starch dissolve?

Boil the mixture in the test tube until a gelatinous mass has been formed. Pour the mass into a beaker of water and stir thoroughly.

Does the boiled starch dissolve?

When starch is heated with water, the cellulose envelope inclosing each starch granule bursts, and a gelatinous mass is formed. On longer heating some of the starch passes into solution.

Half fill a test tube from the beaker containing the starch solution and keep the test tube for part (*d*).

(*b*) Test for starch.

Add a drop of iodine solution to the contents of the beaker.

Result?

(*c*) **Conversion to dextrine.**

Support an evaporating dish in the small ring of a ring-stand. Place one third of a test-tubeful of starch in the dish, having it well above the small flame of a bunsen burner (Figure 56). The starch should be heated just hot enough to turn it barely yellow, but not hot enough to char it. The temperature of conversion from starch to dextrine is between 200° and 250° C.

Figure 56.

Continue the heating for fifteen minutes, stirring meanwhile with a glass rod; then allow the dish to cool. Add a little water to the mass in the dish, and stir the mixture. Rub some of the mass between the fingers.

What characteristic has it?

This is due to the fact that some of the starch has been converted into dextrine.

Why is dextrine used in making mucilage and other adhesives?

Pour a few drops of the liquid in the evaporating dish into a test tube two thirds filled with water, and shake the test tube. Then add a drop of iodine solution. The violet to red color produced is characteristic of dextrine.

(*d*) **Test for glucose.**

Dissolve a little glucose in 10 cc. of water. Heat in a second test tube 10 cc. of Fehling's solution to the boiling point. To this hot liquid add a little of the glucose solution.

Result?

To some of the starch solution kept from part (*a*), add 10 cc. of the hot Fehling's solution.

Result?

(*e*) **Conversion of starch to glucose.**

Put a little starch into a third of a test tube of water and then add three or four drops of hydrochloric acid. Boil the mixture for ten minutes.

Test the resulting liquid with Fehling's solution.

Result?

What change has occurred?

EXPERIMENT 82

Food Constituents. Part I

Fats. Protein

APPARATUS. Test tubes; bunsen burner; ring-stand with one large and one very small ring; two porcelain evaporating dishes; beaker, 150 cc.

MATERIAL. Benzol; filter paper; Geis's biuret reagent, made by adding 3 per cent copper sulphate solution to 10 per cent potassium hydroxide solution a drop at a time, until a faint but perceptible blue color is imparted to the resulting solution; foods for testing, such as fat meat, flour, milk, nuts, sugar, peas, etc.

Test several foods as directed below, and fill in the tabular form.

(*a*) Test for oil or fat.

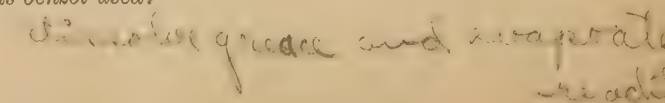
Divide as finely as possible the sample to be tested. Use enough to fill a test tube to the depth of one half to three fourths of an inch. Add benzol to cover the solid to the depth of one half inch.

Caution! Benzol is inflammable.

Warm the contents gently by standing the test tube in a beaker of hot water. Allow the test tube to stand for five minutes, with occasional shaking and warming.

Fold a filter paper as for a funnel. Suspend it in a small ring in the ring-stand. Pour a few drops of the liquid from the test tube into the point of the filter paper, and allow the benzol to evaporate completely. If the food contains oil or fat, more or less of a line of grease will be left around the stained spot on the paper. This can be more easily seen if the paper is held to the light; the grease spot then appears lighter than the rest of the paper.

Why was benzol used?



Why was the sample of food divided as finely as possible?

(*b*) **Test for protein.**

Use the food either as a solution or in as finely divided state as possible. Put it into an evaporating dish, and add 5 cc. of Geis's biuret reagent. Put the same quantity of the reagent into a second evaporating dish for comparison in detecting a change of color. If the food contains protein matter, the solution in the first dish will acquire a pink or violet color. The change will be readily seen if the two dishes are compared.

Test several foods and record your results in the table.

Which of the foods contained no protein?

TABLE

Food Tested	Fat Present or Absent?	Protein Present or Absent?
peanut	yes	
flour	no	

EXPERIMENT 83

Food Constituents. Part II

Carbohydrates

APPARATUS. Test tubes; bunsen burner.

MATERIAL. Starch; glucose; cane sugar; fruit; potato; flour; meat; beets; potassium iodide solution of iodine; Fehling's solution (purchase as two solutions and mix just before using); hydrochloric acid Molisch's reagent (15 per cent solution of α-naphthol in alcohol); concentrated sulphuric acid.

(*a*) Test for carbohydrates. (Molisch test.)

To 5 cc. of a solution obtained by boiling the ground food with water for several minutes, add 2 drops of a solution of α-naphthol (15 per cent solution in alcohol). Hold the tube containing this mixture in an inclined position, and add slowly 3 cc. of concentrated sulphuric acid in such a manner that it slides down the side of the tube and makes a separate layer below the mixture already present in the tube. A purple ring will form between the two layers if the original solution contained any carbohydrate.

Result?

(*b*) Test for starch.

To a pinch of starch add 5 cc. of water and boil the mixture in a test tube till a gelatinous mass has been formed. Dilute this by nearly filling the tube with water. Add a drop of iodine solution.

Result?

(*c*) **Test for glucose.**

Dissolve a little glucose in 10 cc. of water. Heat in a second test tube 10 cc. of Fehling's solution to the boiling point. To this hot liquid add a little of the solution of glucose.

Result?

(*d*) **Formation of glucose from starch and cane sugar.**

To about 1 gram of the starch add 10 cc. of water and 3 or 4 drops of hydrochloric acid. Boil the mixture for ten minutes. Test the resulting solution for glucose.

Result?

In a similar way, boil cane sugar solution with hydrochloric acid and test the product for glucose.

Result?

Under what circumstances can Fehling's solution be used as a test for cane sugar?

(*e*) **Test of samples.**

Test the samples of food given you, first for carbohydrates in general, and then for starch and glucose. If a food contains neither of these, test it for cane sugar. Use the food in a finely divided state, and boil about 1 gram of it with 10 cc. of water for five minutes before making the test.

Record your results in the table on page 230.

Why can not the test for cane sugar be used in the presence of starch?

TABLE

Food Tested	Starch Present or Absent?	Glucose (or Cane Sugar) Present or Absent?

EXPERIMENT 84

Constituents of Milk

APPARATUS. Graduate, 100 cc.; beakers, 100 cc., 250 cc., 500 cc.; watch glass to cover 250 cc. beaker; funnel; glass stirring rod; porcelain crucible, # 0; pipe-stem triangle; wire gauze with asbestos center; ring-stand with two rings; bunsen burner; test tube; two evaporating dishes.

MATERIAL. Sample of sweet milk; acetic acid, 10 cc. glacial acetic acid in 740 cc. of water; sodium hydroxide solution, 4 grams of NaOH per liter; piece of blotting paper a little smaller in diameter than the watch glass; filter paper to fit funnel; carbon tetrachloride; Geis's biuret reagent; Fehling's solution; milk sugar; distilled water.

(*a*) **Casein.**

Pour 25 cc. of milk into a 500 cc. beaker and add 175 cc. of distilled water. Now add, a drop at a time with constant stirring, 40 cc. of the solution of acetic acid. Allow the mixture to stand for about 20 minutes, meanwhile proceeding with part (*b*). Then filter the milk. Save the filtrate for use in part (*c*).

Why does milk curdle when it enters the stomach?

acetic acid.

Put the paper and contents into a beaker of water and rinse the precipitate from the paper. The precipitate is casein. Test this for protein by the method given in Experiment 82.

Result? yes a little

Allow the casein to settle, then pour off the liquid and add to the casein 100 cc. of the sodium hydroxide solution. If the casein does not dissolve, add a little more of the alkali.

How could casein be reprecipitated from this solution?

(*b*) **Fat.**

Half fill a 250 cc. beaker with water. Set it on a piece of wire gauze placed on the ring of a ring-stand, and heat the water. As soon as the water commences to boil, lower the flame so it will furnish only enough heat to keep the water boiling gently. While waiting for the water to boil, wet the piece of blotting paper with milk by pouring a thoroughly mixed sample down a stirring rod so that it will drop on the paper in such a manner as to distribute the milk evenly over the surface of the paper. Put the paper wet with milk on the watch glass, set the glass on the beaker of boiling water, and allow it to remain until the milk has evaporated to dryness.

Remove the watch glass containing the paper and allow it to cool. When the glass is cold, pour into it sufficient carbon tetrachloride to cover the paper. Work the paper in the liquid by means of a stirring rod for two or three minutes, then remove the paper and evaporate the carbon tetrachloride over steam.

What remains on the watch glass?

(*c*) **Lactalbumen.**

Heat the filtrate obtained in part (*a*) to boiling and continue the boiling until a clear fluid can be obtained by filtration. Save this filtrate for use in part (*d*). The residue is lactalbumen. Determine whether it is a protein.

Result?

Casein does not coagulate when milk is boiled.

What is the scum that appears on the surface of boiling milk?

(*d*) **Sugar.**

Fill one third of a test tube with the filtrate obtained in part (*c*), add sufficient Fehling's solution to have the tube half full

of the mixture, and heat the contents of the tube to boiling. Lactose, milk sugar, reduces Fehling's solution in a manner similar to glucose. See Experiment 83.

Examine a sample of milk sugar.

How does its sweetness compare with that of sugar?

(*e*) **Ash.**

Evaporate 5 cc. of milk to dryness in a porcelain crucible placed on a pipe-stem triangle supported on the rim of a beaker containing boiling water. Dry the crucible, then gradually raise the temperature to the full capacity of your bunsen burner. Continue the heat until the carbon has been completely burned and only a white ash remains.

Would milk sugar leave an ash?

APPENDIX

APPENDIX

I. PHYSICAL CONSTANTS

OF THE IMPORTANT ELEMENTS

Approximate Weights Should Be Used in All Calculations, Except Those of Equivalents

Element	Symbol	Atomic Weights		Valence	Specific Gravity		Melting Point	Boiling Point
		Approximate	Exact O = 16		Water = 1	Air = 1	°C.	°C.
Aluminum	Al	**27**	27.1	III	2.7		657	2200
Antimony	Sb	**120**	120.2	III V	6.6		630	1600
Argon	A	**40**	39.88	——		1.38	− 188	− 186
Arsenic	As	**75**	74.96	III V	5.7		. . .	<449 sublimes
Barium	Ba	**137**	137.37	II	3.8		850	950
Bismuth	Bi	**208**	208.0	III V	9.7		269	1435
Boron	B	**11**	11.0	III	2.4		infusible	3500
Bromine	Br	**80**	79.92	I	3.1		− 7.3	59 sublimes
Cadmium	Cd	**112**	112.4	II	8.6		321	778
Calcium	Ca	**40**	40.07	II	1.5		about 805	. . .
Carbon	C	**12**	12.005	IV	amorphous 1.7–2.1		infusible	3500
Chlorine	Cl	**35.5**	35.46	I		2.49	− 102	− 33.6
Chromium	Cr	**52**	52.0	II III VI	6.9		1505	2200
Cobalt	Co	**59**	58.97	II	8.7		1490	. . .
Copper	Cu	**63.6**	63.57	I II	8.9		1083	2310
Fluorine	F	**19**	19.0	I		1.31	− 223	− 187
Gold	Au	**197**	197.2	I III	19.3		1062	2530
Helium	He	**4**	4.00	——		0.14	− 269	− 268.5
Hydrogen	H	**1**	1.008	I		0.07	− 259	− 252
Iodine	I	**127**	126 92	I	4.9		114	184
Iron	Fe	**56**	55.84	II III	7.9		1530	2450
Lead	Pb	**207**	207.2	II IV	11.3		327	1525

Element	Symbol	Atomic Weights		Valence	Specific Gravity		Melting Point	Boiling Point
		Approximate	Exact O = 16		Water = 1	Air = 1	°C.	°C.
Lithium	Li	**7**	6.94	I	0.53		186	<1400
Magnesium	Mg	**24**	24.32	II	1.7		650	1120
Manganese	Mn	**55**	54.93	II IV	7.4		1225	1900
Mercury	Hg	**200**	200.6	I II	13.6		− 38.8	357
Nickel	Ni	**58.7**	58.68	II	8.7		1450	. . .
Nitrogen	N	**14**	14.01	III V		0.97	− 213	− 195
Oxygen	O	**16**	16.00	II		1.10	<−227	− 183
Phosphorus	P	**31**	31.04	III V	1.8 white		44.1 white	290
Platinum	Pt	**195**	195.2	IV	21.2		1753	. . .
Potassium	K	**39**	39.10	I	0.87		62.5	757
Silicon	Si	**28**	28.3	IV	2.4		1450	3500
Silver	Ag	**108**	107.88	I	10.5		961	1955
Sodium	Na	**23**	23.0	I	0.97		97.6	877
Strontium	Sr	**87**	87.63	II	2.5		900	. . .
Sulphur	S	**32**	32.06	II IV VI	2.0		114.5 rhombic	444.6
Tin	Sn	**119**	118.7	II IV	7.3		232	1450–1600
Zinc	Zn	**65**	65.38	II	7.1		419	918

II. TABLE OF SOLUBILITIES

S = soluble in water. **I** = insoluble in water. **P** = slightly (partly) soluble in water.
Ia = insoluble in water and dilute acids.

	Aluminum	Ammonium	Antimony	Arsenious	Barium	Bismuth	Cadmium	Calcium	Cobalt	Copper	Ferrous (Fe^{++})	Ferric (Fe^{+++})	Lead	Magnesium	Manganese	Mercurous (Hg^{+})	Mercuric (Hg^{++})	Nickel	Potassium	Sodium	Silver	Zinc
Acetate	S	S	—	—	S	—	S	S	S	S	S	—	S	S	S	P	S	S	S	S	S	S
Bromide	S	S	P	S	S	P	S	S	S	S	S	S	P	S	S	I	S	S	S	S	Ia	S
Carbonate	—	S	—	—	I	I	I	I	I	I	I	—	I	I	I	I	I	I	S	S	I	I
Chlorate	S	S	—	—	S	—	S	S	S	S	—	—	S	S	—	S	S	—	S	S	S	S
Chloride	S	S	P	S	S	P	S	S	S	S	S	S	P	S	S	I	S	S	S	S	Ia	S
Chromate	—	S	—	—	I	I	—	I	I	I	—	—	Ia	S	—	P	P	—	S	S	I	—
Hydroxide	I	S	P	S	S	I	I	P	I	I	I	I	I	I	I	—	I	I	S	S	—	I
Iodide	S	S	P	P	P	I	S	S	S	—	S	—	I	S	S	I	I	S	S	S	Ia	S
Nitrate	S	S	—	—	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Oxide	I	—	I	P	S	I	I	P	I	I	I	I	I	I	I	I	I	I	S	S	I	I
Phosphate	I	S	—	—	I	I	I	I	I	I	I	I	I	I	P	I	I	I	S	S	I	I
Sulphate	S	S	—	—	Ia	S	S	P	S	S	S	S	Ia	S	S	P	S	S	S	S	P	S
Sulphide	—	S	Ia	Ia	S	Ia	Ia	P	I	Ia	P	—	Ia	S	I	Ia	Ia	I	S	S	Ia	I
Sulphite	—	S	—	—	I	—	S	P	I	S	P	—	I	P	—	—	—	I	S	S	S	P

III. GENERAL RULES FOR SOLUBILITY

Certain generalizations can be made concerning compounds shown in the table on the opposite page. The exceptions to these generalizations are few and unimportant for the beginner.

1. All *sodium*, *potassium*, and *ammonium* compounds are *soluble* in water.
2. All *nitrates*, *chlorates*, and *acetates* are *soluble* in water.
3. All *chlorides* are *soluble*, except those of silver, mercury (mercurous), and lead (lead slightly soluble).
4. All *sulphates* are *soluble*, except those of barium, lead, and calcium (calcium slightly soluble). The silver and the mercurous sulphates are only moderately soluble.
5. All *carbonates* are *insoluble*, except those of sodium, potassium, and ammonium.
6. All *oxides* and *hydroxides* are *insoluble*, except those of ammonium, sodium, potassium, and barium; calcium hydroxide is slightly soluble.

IV. VOLATILITY OF COMPOUNDS THAT MAY RESULT FROM DOUBLE DECOMPOSITIONS

1. Compounds volatile at ordinary temperatures:

$$HCl \quad HBr \quad HF \quad H_2S$$

2. Compounds decomposing at ordinary temperatures yielding volatile products:

$$H_2CO_3 \ (H_2O + CO_2)$$
$$H_2SO_3 \ (H_2O + SO_2)$$
$$NH_4OH \ (H_2O + NH_3)$$

3. Compounds volatile at varying temperatures below 338° (boiling-point of sulphuric acid):

	Boiling-point		Boiling-point
H_2O,	100°	HNO_3,	86°
HCl (aqueous solution),	110°	HNO_2 (aqueous solution),	120°
HBr (aqueous solution),	126°	$HC_2H_3O_2$,	118°

V. APPROXIMATE WEIGHT OF ONE LITER OF COMMON GASES UNDER STANDARD CONDITIONS

Acetylene,	1.17 grams	Hydrogen sulphide,	1.53 grams
Ammonia,	0.77 "	Marsh gas,	0.72 "
Carbon dioxide,	1.98 "	Nitrogen,	1.26 "
Carbon monoxide,	1.26 "	Nitric oxide,	1.35 "
Chlorine,	3.20 "	Nitrous oxide,	1.98 "
Hydrogen chloride,	1.64 "	Oxygen,	1.43 "
Hydrogen,	0.09 "	Sulphur dioxide,	2.88 "

VI. THE METRIC SYSTEM AND ITS EQUIVALENTS

A. Fundamental Units

The *International Standard Meter* is the distance between two lines, at 0° Centigrade, on a platinum-iridium bar deposited at the International Bureau of Weights and Measures near Paris, France.

MEASURES OF LENGTH

10 millimeters (mm.) = 1 centimeter (cm.)
10 centimeters = 1 decimeter (dm.)
10 decimeters = 1 meter (m.)

The *International Standard Kilogram* is the weight of a mass of platinum-iridium deposited at the International Bureau of Weights and Measures.

The *liter* is equal to a cubic decimeter and it is measured by the quantity of distilled water which, at its maximum density (4° Centigrade), will counterpoise the standard kilogram.

Since *a liter contains 1000 cubic centimeters*, one cubic centimeter of water, at 4° Centigrade, weighs 1 gram.

B. Important Metric Relations

Measure	Relation
Linear	millimeter = 0.001 meter centimeter = 0.01 meter decimeter = 0.1 meter
Capacity	cubic centimeter = 0.001 liter
Weight	milligram = 0.001 gram centigram = 0.01 gram decigram = 0.1 gram
	kilogram = 1000 grams

C. Comparison of Metric with other Weights and Measures

Metric Unit	Equivalent		Measure
	Approximate	Exact	
Meter	. . . .	39.37 inches	U.S. Linear
Centimeter	**0.4** inch	0.3937 inch	U.S. Linear
Liter	**1.06** quarts	1.05668 quarts	U.S. Liquid
Kilogram	**2.2** pounds	2.20462 pounds	U.S. Avoirdupois
Gram	**15.4** grains	15.43235 grains	U.S. Avoirdupois

Unit	U.S. Measure	Equivalent	
		Approximate	Exact
Yard	Linear	**0.9** meter	0.914402 meter
Inch	Linear	**2.5** centimeters	2.54001 centimeters
Gallon	Liquid	**3.8** liters	3.78543 liters
Quart	Liquid	**0.95** liter	0.94636 liter
Fluid ounce	Liquid	**29.6** c.c.	29.574 c.c.
Pound	Avoirdupois	**0.45** kilogram	0.45359 kilogram
Pound	Avoirdupois	**453.6** grams	453.59 grams
Ounce	Avoirdupois	**28.3** grams	28.3495 grams
Grain	Avoirdupois	**0.06** gram	0.0648 gram

VII. PRESSURE OF WATER VAPOR, OR AQUEOUS TENSION

(In Millimeters of Mercury)

Temperature	Pressure	Temperature	Pressure
0.0° C.	4.6 mm.	21.5° C.	19.1 mm.
5	6.5	22	19.7
10	9.2	22.5	20.3
10.5	9.5	23	20.9
11	9.8	23.5	21.5
11.5	10.1	24	22.2
12	10.5	24.5	22.9
12.5	10.8	25	23.5
13	11.2	25.5	24.3
13.5	11.6	26	25.0
14	11.9	26.5	25.7
14.5	12.3	27	26.5
15	12.7	27.5	27.3
15.5	13.1	28	28.1
16	13.6	28.5	28.9
16.5	14.0	29	29.8
17	14.4	29.5	30.7
17.5	14.9	30	31.6
18	15.4	40	55.0
18.5	15.9	50	92.2
19	16.4	60	149.2
19.5	16.9	70	233.8
20	17.4	80	355.5
20.5	17.9	90	526.0
21	18.5	100	760.0

LIST OF SUPPLIES

THE following list is an estimate of the material advisable to purchase for a class of ten pupils, provided each student performs all of the experiments described. It names a generous supply of apparatus and chemicals, allowing for a reasonable amount of breakage.

In case of somewhat expensive apparatus it has been assumed that two pupils will use one piece of apparatus. Provision has been made for schools not having laboratories equipped for the use of the electric current.

In many schools it is practicable to arrange the laboratory work so that all of the members of the class do not perform a certain experiment at the same time. In such cases, the number of expensive pieces of apparatus required may be less than that mentioned in the list.

An asterisk follows certain items on the list. Such articles are relatively less necessary, as they are usually needed for only a single experiment.

GENERAL APPARATUS

5 pieces apparatus, electrolytic.*
Form shown in Fig. 10.

1 apparatus, electrolytic, carbon electrodes.*
Form shown in Fig. 24.

1 doz. sheets asbestos, thin (baking-sheet).*

10 squares asbestos, 6″ × 6″.

2 balances, platform, with weights for weighing from 1000 grams to 0.1 gram.

10 balances, horn pan, $7\frac{1}{2}$″ beam, with weights for weighing from 100 grams to 10.01 gram.

1 barometer.

10 blowpipes, 8″.
10 brushes, test-tube.
10 brushes, small tube.
10 burners, bunsen.
20 candles, birthday.
10 capsules, brass, with wire holder and brass ramrod, for holding sodium below water.*

These can be obtained from Eimer and Amend, New York.

15 cells, dry battery.*
2 pkg. cigar lighters, wood.
20 clamps, iron, small; for test tubes, burettes, etc.
10 clamps, iron, large; for Liebig condensers.
1 gross corks, assorted sizes, long.
1 set cork borers, 6 in set.
12 crucibles, porcelain, with lids, #00.
25 cups, agate ware, 1 pt. (or stew pans).*
12 dishes, porcelain, evaporating, #0.
10 dishes, porcelain, evaporating, #1.
10 droppers, medicine.
10 files, triangular, 5″.
10 pr. forceps, iron, 4″.
10 squares gauze, iron wire with asbestos center, 5″ × 5″.
1 glass cutter.
10 holders, test tube.
5 lamps, incandescent, 100 watt.*
5 magnifiers, Coddington lens, or other make.
5 mortars, with pestle, $3\frac{1}{2}$″.
12 pans, enamel, shallow, 1 qt.
10 pans, iron, 5 in., shallow form, "sand bath."
10 pkg. paper, filter, qualitative, good quality, 4″.
2 sheets paper, black, glazed.
10 pinch-cocks, Mohr's, medium.*
10 ft. platinum wire, #25.*
10 racks, test tube, for 12 tubes.
5 shears, 6″.*
5 sheets sandpaper, #1.
5 spatulas, horn, 6″.

10 spoons, deflagration, diam. of bowl 1 cm.
10 stands, iron, ring, 3 rings.
2 lb. stoppers, rubber, assorted sizes, #0–5, one- and two-hole.
1 pkg. tapers, wax.
10 thermometers, chemical, 0°–250° C.
1 spool thread, #50, cotton.
10 tripods, iron, for supporting dishes over burner, ring 4″ in diam.
10 triangles, pipestem, size to support #00 porcelain crucible.
10 troughs, pneumatic.
50 ft. tubing, rubber, inside diam. $\frac{3}{16}$″.
20 ft. tubing, rubber, inside diam. $\frac{3}{8}$″.
5 yard-sticks.*

GLASSWARE

24 beakers, 100 cc.
36 beakers, 150 cc.
36 beakers, 250 cc.
12 beakers, 500 cc.
100 bottles, reagent, 4 oz.
30 bottles, wide mouth, 4 oz.
40 bottles, wide mouth, 6 oz., with two-hole rubber stoppers to fit.
10 bottles, wide mouth, 8 oz., with two hole rubber stoppers to fit.
2 bottles, wide mouth, 16 oz., with two-hole rubber stoppers to fit.
10 bottles, acid, $2\frac{1}{2}$ liters.
20 burettes, 50 cc., graduated to $\frac{1}{10}$ cc., complete.*
10 condensers, Liebig, 15″.*
12 flasks, distilling, 250 cc.*
12 flasks, Florence, 50 cc.
12 flasks, Florence, 100 cc., with one- and two-hole rubber stoppers to fit.
12 flasks, Florence, 250 cc., with two-hole rubber stoppers to fit.
12 flasks, Erlenmeyer, 50 cc.
12 flasks, Erlenmeyer, 250 cc., with two-hole rubber stoppers to fit.
24 funnels, accurate 60°, $2\frac{1}{2}$″.
10 graduates, 50 cc., graduation marks to 1 cc.
2 graduates, 1000 cc., graduation marks to 10 cc.
10 jars, battery, about 4″ × 5″.
10 jars, hydrometer, 12″ × 2″.

5 pipettes, 10 cc.*
40 plates, glass, $2\frac{1}{2}'' \times 2\frac{1}{2}''$.
10 plates, glass, $4'' \times 4''$.
40 plates, cobalt glass, for flame tests, $3'' \times 2''$.*
15 retorts, tubulated, with ground-glass stopper, 4 oz.*
1 lb. rods, 3 mm. diameter.
10 tubes, gas measuring, 50 cc., graduated to $\frac{1}{10}$ cc.
15 tubes, test, hard glass, for ignition, $6'' \times \frac{5}{8}''$, with one-hole cork stoppers to fit.
12 tubes, test, side arm, 6".*
15 doz. tubes, test, soft glass, medium walls, for heating, $6'' \times \frac{3}{4}''$.
2 doz. tubes, test, soft glass, medium walls, for heating, $4'' \times \frac{1}{2}''$.
15 tubes, thistle, 10", stem $\frac{3}{16}''$ in diameter.
12 tubes, U, 6", with two-hole rubber stoppers to fit.
12 tubes, U, 4", with one-hole rubber stoppers to fit.*
2 lb. tubing, soft glass, medium walls for bending, outside diameter 4 mm.
24 watch glasses, diameter $2\frac{1}{2}''$.
40 watch glasses, Syracuse form, diameter 3".*

CHEMICALS AND OTHER SUPPLIES

1 lb. acid, acetic, 30 %, c. p.
1 oz. acid, acetic, glacial.*
2 oz. acid, boric, c. p.
1 oz. acid, citric, c. p.
4 oz. acid, formic.*
12 lb. acid, hydrochloric, c. p., sp. gr. 1.19.
7 lb. acid, nitric, c. p., sp. gr. 1.42.
2 gr. acid, picric.*
1 lb. acid, oxalic, cryst., c. p.
1 oz. acid, salicylic.*
9 lb. acid, sulphuric, c. p., sp. gr. 1.84.
1 oz. acid, tannic, c. p.
4 oz. acid, tartaric.
2 qt. alcohol, ethyl, 95 %.
1 pt. alcohol, methyl (wood alcohol).*
4 oz. alizarine, paste, 25 %.*

1 oz. alpha naphthol.*
8 oz. aluminum sulphate, cryst.
1 lb. ammonium chloride, c. p.
10 lb. ammonium hydroxide, c. p., sp. gr. 0.9.
8 oz. ammonium nitrate, cryst., c. p.
2 oz. ammonium oxalate, cryst., c. p.*
4 oz. ammonium sulphate, c. p.
1 oz. antimony, lumps (powder, if necessary, just before using).*
1 lb. barium chloride, cryst., c. p.
4 oz. barium nitrate, c. p.
2 oz. benzol.*
5 gr. Biebricht scarlet (or tartrazine, or acid blue).*
8 oz. bleaching powder.
1 lb. boneblack.
1 oz. bromine.*
1 oz. cadmium nitrate, c. p.*
$\frac{1}{2}$ lb. calcite.*
5 lb. calcium carbonate, marble chips.
1 lb. calcium chloride, granular, for drying tubes.
2 oz. calcium nitrate, c. p.
2 lb. calcium oxide, good quality of lime in tin can.
2 oz. calcium phosphate (monocalcium).*
5 lb. calcium sulphate, plaster of Paris, fine.
1 lb. carbon disulphide.
4 oz. carbon tetrachloride.*
$\frac{1}{2}$ lb. chalk, precipitated.*
12 blocks charcoal, for blowpiping.
1 oz. charcoal, wood, powdered.
1 lb. chloroform.*
2 oz. chromium sulphate, c. p. (or chrome alum).
$\frac{1}{2}$ yd. cloth, calico, bleachable color, for bleaching with chlorine.
1 yd. cloth, cotton, bleached, fine goods.
$\frac{1}{8}$ yd. cloth, woolen.*
2 yd. cheesecloth.
1 oz. cobalt nitrate, cryst., c. p.
10 grams congo red.*
4 oz. copper foil, $\frac{1}{100}''$ thick.*

10 sq. in. copper gauze, 80 meshes to inch.*
2 lb. copper rivets, $\frac{1}{2}''$.
$\frac{1}{2}$ lb. copper sheet, $\frac{1}{64}''$ thick.
2 lb. copper turnings, clean, fine.
1 lb. spool copper wire, # 16.
1 lb. spool copper wire, # 18.
1 lb. spool copper wire, # 24.
1 lb. spool copper wire, # 30.
1 oz. copper oxide, powdered, c. p.
1 oz. copper oxide, wire form.
1 oz. copper sulphate, anhydrous.
1 lb. copper sulphate, cryst., c. p.
1 oz. cotton, absorbent.
2 lb. cotton waste, unbleached.*
2 oz. Fehling's solution, two solutions in separate bottles.
10 grams fuchsine.*
8 oz. glucose.*
4 oz. hydrochinone.*
1 lb. hydrogen peroxide.
1 oz. iodine, resublimed.*
10 oz. iron and ammonium citrate, green (ferric ammonium citrate).
4 oz. iron chloride, ferric, c. p.
2 oz. iron filings, fine, clean.
1 lb. iron sulphate, cryst., c. p.
4 oz. iron sulphide, ferrous, in sticks, for H_2S.
1 lb. spool iron wire, # 16.
1 lb. spool iron wire, # 25.
2 oz. lead acetate.
1 lb. lead nitrate, c. p.
5 lb. lead shot, # 10.
$\frac{1}{2}$ oz. lithium nitrate.
1 oz. litmus cubes.
$\frac{1}{2}$ quire each, red and blue litmus paper.
4 oz. logwood, ground.*
1 oz. magnesium, powder.*
1 oz. magnesium, ribbon.
1 lb. magnesium sulphate, cryst.

10 grams malachite green.*
2 lb. manganese dioxide, fine, granular, free from carbon.
4 oz. manganese dioxide, c. p.*
1 oz. mercuric nitrate.
8 oz. mercuric oxide, red.
1 oz. mercurous nitrate.
1 pt. molasses, good quality, kettle-rendered.*
24 sheets paper, unglazed.
10 grams phenolphthalein.
½ oz. phosphorus, red.
1 oz. phosphorus, yellow.
1 lb. potassium and aluminum sulphate, alum.
1 oz. potassium antimonyl tartrate (tartar emetic).*
4 oz. potassium acid tartrate.
10 oz. potassium bromide.
1 lb. potassium chlorate, cryst., c. p.
2 oz. potassium chromate.*
1 oz. potassium and chromium sulphate (chrome alum).*
8 oz. potassium dichromate.*
1 lb. potassium ferricyanide.
2 oz. potassium ferrocyanide.
2 lb. potassium hydroxide, c. p., by alcohol.
2 oz. potassium iodide, c. p.
2 lb. potassium nitrate, cryst., c. p.
1 oz. potassium permanganate, c. p.
1 oz. potassium oxalate.*
8 oz. potassium sulphate, c. p.
1 lb. salt, rock.*
4 oz. silver nitrate, c. p.
1 oz. soap, castile, powdered.
2 oz. sodium.
4 oz. sodium acetate.
2 oz. sodium aluminum sulphate (sodium alum).*
1 lb. sodium bicarbonate, baking soda.
4 oz. sodium bisulphite.*
1 lb. sodium carbonate, cryst., washing soda.
8 oz. sodium carbonate, pure, dry.

5 lb. sodium chloride, salt, fine.
2 oz. sodium chromate, powdered.*
1 lb. sodium hydroxide, c. p., by alcohol.
1 oz. sodium iodide (or potassium iodide).*
1 lb. sodium nitrate, c. p.
2 oz. sodium peroxide.
8 oz. sodium phosphate, c. p.
2 oz. sodium phosphate (monosodium).*
2 lb. sodium sulphate, cryst.
1 lb. sodium sulphite, pure, dry.*
2 lb. sodium thiosulphate ("hypo").
1 lb. sodium tetraborate (borax).
1 lb. starch, corn.
1 lb. starch, potato.
1 oz. strontium nitrate, c. p.
2 lb. sulphur, roll.
8 oz. tin, granulated.
1 sheet turmeric paper.
1 qt. vinegar, cider.*
1 oz. wool, glass, fine Bohemian.
1 pkg. wool, steel, fine.
8 oz. yarn, woolen, white.*
½ lb. zinc, sheet.
2 lb. zinc, granulated (mossy).
2 oz. zinc dust.*
1 oz. zinc nitrate.
8 oz. zinc sulphate.

A very few articles, such as flour, yeast, foods for testing, and other articles of common household use, have been omitted from the above list.